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Volume 28 October 1, 1945 Number 10

EFFECT OF MIGRATION OF CLAY MINERALS AND HYDROUS ALUMINUM OXIDES ON THE COMPLEXITY OF CLAY*

By Victor T. Allen

Abstract

Movement of montmorillonite, nontronite, kaolinite, halloysite, dickite, gibbsite, and diaspore within clays after they were first formed is indicated by the relations reported in this paper. Migration of these minerals takes place rarely by transfer of their constituents as true solutions but generally as colloidal suspensions whose formation and movement are favored by conditions of good drainage and the presence of dispersing agents. This migration increases the complexity of the clays affected by it and accounts for some of the variations in the composition of a single body of clay met by the clay producer.

I. Introduction

The formation of clay minerals and hydrous aluminum oxides by weathering processes operating in place to produce residual decomposition products is well established. The purpose of this paper is to report clay minerals and hydrous aluminum oxides observed in situations where the evidence indicates their migration or movement within the rock or their introduction from the outside. This migration increases the complexity of the clays affected by it.

Two modes of transportation of clay minerals are suggested for the secondary relationships which have been observed, namely, (1) migration in colloidal suspensions and (2) transfer of their constituents in true solution. Some examples of each will be considered before taking up the petrographic descriptions of the secondary occurrences of clay minerals and hydrous aluminum oxides.

The writer's interest in the secondary redistribution of clay minerals began in 1929 when he observed a brown clay mineral in the weathered zones of glacial deposits filling cracks, rootlet tubes, and cavities with its cleavage plates parallel to the sides of the openings. When plates of this clay mineral were finely ground and shaken in water, they remained in suspension for a long time. When a glass slide was arranged to catch the falling particles, the plates of the clay mineral arranged themselves with their cleavage surfaces parallel to the surface of the slide. After carefully siphoning off the water and allowing the clay mineral to dry, it was found that an optical interference figure with a small optic angle could be obtained from any part of the slide, provided the clay mineral sheets had the proper thickness. This indicated that minute particles of this clay mineral in a colloidal suspension could arrange themselves under the right conditions to form a larger plate with sufficient optical continuity that it acted as a single crystal plate. The ease with which a colloidal suspension of it could be formed also suggested that the plates of this clay mineral occurring along former openings in the weathered zones of glacial deposits had been carried and deposited there in colloidal suspensions under conditions of good drainage.[1] Under conditions of poor drainage, the clay minerals remain in the zone close to the surface in which they are developed by weathering and form a plastic clay which has been termed gumbotil.[2] Broad, flat upland areas are underlain just below their surfaces by gumbotil; on hill slopes or rolling topography, however, the equivalent position below the surface is not occupied by gumbotil but by a silty zone from which significant amounts of clay minerals have been carried to a lower zone. It appears, therefore, that the amount of ground water which circulates through a clay under conditions of good drainage determines whether the clay minerals remain in place or are carried to a new location. The process is especially effective when ground water con-

* Supplementary title for Forty-Seventh Annual Program, No. 7, *Bull. Amer. Ceram. Soc.*, **24** [5] 180 (1945).
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[1] (*a*) V. T. Allen, "Petrography of Weathered Zones of Glacial Deposits," *Bull. Geol. Soc. Amer.*, **41** [1] 85–86 (1930).
(*b*) V. T. Allen, "Petrographic and Mineralogical Study of Underclays of Illinois Coal," *Jour. Amer. Ceram. Soc.*, **15** [10] 564–73 (1932).

[2] M. M. Leighton and Paul MacClintock, "Weathered Zones of Drift-Sheets of Illinois," *Jour. Geol.*, **38** [1] 28–53 (1930).

tains a dispersing agent which aids in supplying minute mineral particles for colloidal suspensions.

The difficulties of dissolving, transporting, and redepositing the constituents of clay minerals by ground water are so great compared with those involved in colloidal migration that some may ask what is the evidence of their transfer as true solutions. Near Gordon, Georgia, the writer observed in the clays of the Jackson group, fossil shells that were originally calcium carbonate now replaced by montmorillonite, which retains the shell structure. Similar replacement has been discussed by Ross and Stephenson[3] in their description of calcareous shells replaced by beidellite at Pontotoc, Mississippi. The process required the removal of calcium carbonate from the shells and at the same time the introduction of Al_2O_3, SiO_2, and small amounts of Fe_2O_3, MgO, and other elements to form beidellite. The removal of the calcium from the shell was complete since the beidellite with shell structure at Pontotoc contains no calcium, but the beidellite at other localities contains small amounts of calcium. The preservation of the shell structure required a particle-by-particle removal of the dissolved substances by solutions having great penetrative power and mobility. These properties are characteristic of true solutions rather than of colloidal suspensions.

Where clay minerals form part of a paragenetic sequence of minerals, their deposition probably resulted from true solutions because these surpass colloidal suspensions in mobility, in versatility in conveying a number of elements at the same time, and in precipitating them successively as compounds and also in their capacity for keeping the feeding channels open. The probability that true solutions have operated is increased when one or more of them in the sequence consists of a mineral or minerals generally accepted as forming from true solution. For example, the kaolin minerals in veins at Hobart Butte, Oregon, line fractures that are coated with pyrite crystals and scales of siderite of a thickness equal to those of the kaolin minerals; all are associated with realgar (AsS) and arsenates in a way that suggests they were deposited from true solutions at slightly elevated temperatures.

Dickite and kaolinite have been described associated with ore minerals and in geodes where deposition has been attributed[4] to true solution.

Figure 8 (*B*) illustrates a geode of chalcedony and quartz enclosing white clay from St. Louis County, Missouri. Microscopic examination reveals that the clay in the center of the geode consists of tiny hexagonal plates with the extinction angle of dickite. Other geodes of similar appearance from the same locality enclose kaolinite so that the dickite and kaolinite were deposited from similar solutions that probably varied only in temperature.

Alunite, hydrous potassium aluminum sulfate, is commonly associated with kaolin minerals formed by hydrothermal solutions.[5] Alunite filling shatter cracks in halloysite at Huron, Indiana, has been described by Ross and Kerr.[6] Previous investigators agree that the halloysite was introduced by sulfate solutions which resulted from the decomposition of iron sulfide and which obtained the halloysite by reaction with the underlying shale; meteoric waters are regarded here as the active agent in the solution, circulation, and deposition of the halloysite clay. Because the aluminum sulfate was carried from the Chester shale to react with and replace with halloysite the quartz pebbles of the Pottsville sandstone, the transfer was made as a true solution.

Evidence regarding the ease with which colloidal suspensions of gibbsite and diaspore can be formed is lacking. Conditions probably exist under which these minerals can migrate as colloidal suspensions. In addition, there are indications that the constituents of these hydrous aluminum oxides under some conditions are transferred as true solutions. Evidence which is outside of the scope of this paper indicates that silicate minerals can be decomposed chemically and silica removed in solution.[7] According to Roy[8] and others, the silica in natural waters is carried in true solution, probably ionic. If true solutions of the constituents of hydrous aluminum oxides are liberated during chemical decomposition, it appears likely that they would be transported as such rather than changed over to the colloidal state before their deposition in the secondary relationships in which they have been observed.

The primary purpose of this paper is to record evidence that clay minerals and hydrous aluminum oxides have migrated within clays since they were first formed. The writer considers that this migration generally takes place as dispersed systems of tiny mineral particles, but at some deposits the genetic relationships indicate the transfer of their constituents as true solutions.

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[3] C. S. Ross and L. W. Stephenson, "Calcareous Shells Replaced by Beidellite," *Amer. Mineral.*, **24** [6] 393–98 (1939).

[4] (*a*) W. A. Tarr and W. D. Keller, "Dickite in Missouri," *ibid.*, **21** [2] 109–14 (1936).

(*b*) W. A. Tarr and W. D. Keller, "Some Occurrences of Kaolinite Deposited from Solution," *ibid.*, **22** [8] 933–35 (1937); *Ceram. Abs.*, **17** [4] 157 (1938).

[5] (*a*) B. S. Butler, and H. S. Gale, "Alunite, a Newly Discovered Deposit near Marysvale, Utah," *U. S. Geol. Survey Bull.*, No. 511, pp. 1–64 (1912).

(*b*) Whitman Cross, "Alunite of Rosita Hills, Colo.," *ibid.*, No. 511, pp. 38–43 (1912).

(*c*) F. L. Ransome, "Geology and Ore Deposits of Goldfield, Nevada," *U. S. Geol. Survey Prof. Paper*, No. 66, pp. 189–95 (1915).

(*d*) G. F. Loughlin, "Recent Alunite Development near Marysvale and Beaver, Utah," *U. S. Geol. Survey Bull.*, No. 620, pp. 237–70 (1916).

(*e*) Eugene Callaghan, "Preliminary Report on Marysvale Region, Utah," *ibid.*, No. 886d, pp. 91–134 (1938).

[6] C. S. Ross and P. F. Kerr, "Halloysite and Allophane," *U. S. Geol. Survey Prof. Paper*, No. 185G, pp. 140–44 (1934); *Ceram. Abs.*, **14** [4] 101 (1935).

[7] V. T. Allen, "Mineral Composition and Origin of Missouri Flint and Diaspore Clay," *Mo. Geol. Survey and Water Resources, 58th Biennial Rept.*, Appendix IV, pp. 1–24 (1935); *Ceram. Abs.*, **17** [1] 40 (1938).

[8] C. J. Roy, "Silica in Natural Waters," *Amer. Jour. Sci.*, **243** [7] 393–403 (1945).

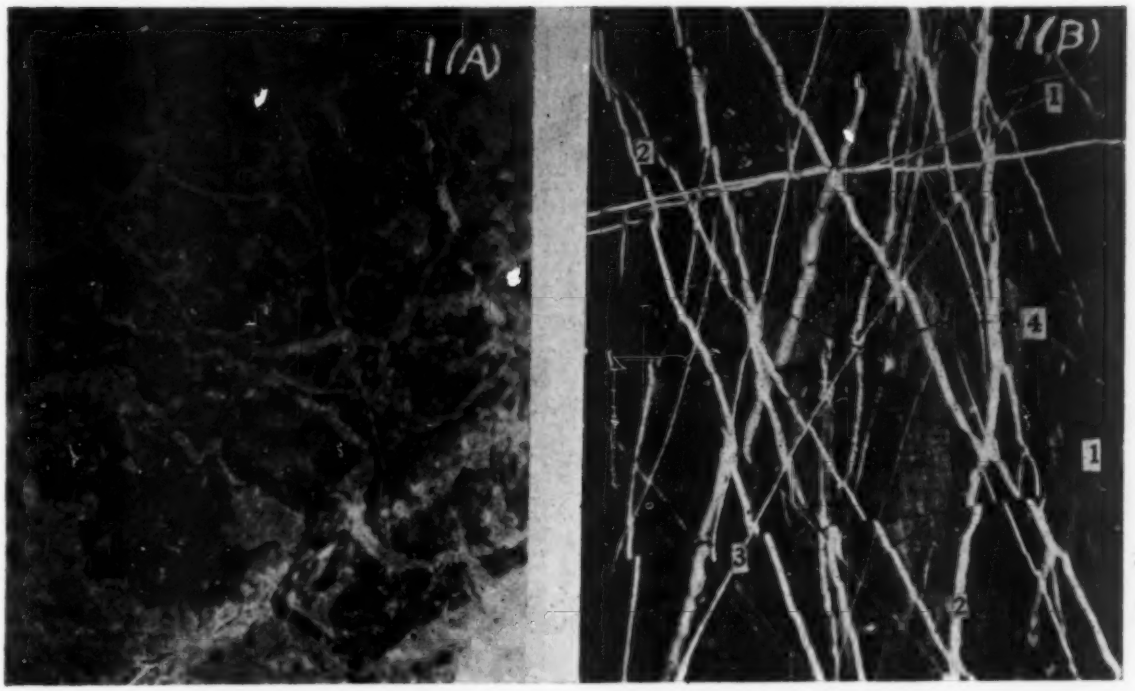

FIG. 1.—(*A*) Veins of white montmorillonite cutting gray or buff montmorillonite, Clayfields, Ga., ×260; (*B*) at least four periods of clay migration indicated by sets of intersecting beidellite-filled fractures, numbered in order of formation, in beidellite clay, Twin Falls, Idaho; crossed nicols, ×150; photograph by C. S. Ross.

II. Migration of Montmorillonite

The extreme thinness of the plates composing the clay minerals of the montmorillonite group and the ease with which a colloidal suspension of them can be formed facilitates their migration. The thin molecular plates, moreover, have a water film between each one and during drying have a strong tendency to be oriented parallel to their cleavage direction and this parallelism often aids in recognizing secondary montmorillonite.

At Clayfields, Wilkinson County, Georgia, montmorillonite clays belong to the Jackson group of Eocene age and overlie the important commercial sedimentary kaolin of the Tuscaloosa formation of Upper Cretaceous age. Repeated wetting and drying of the clay has resulted in the formation of concentric cracks around original depositional areas having less shrinkage than the adjoining concentric zone. A complex network of irregular clay veins cuts across early structures. Nearly white montmorillonite fills and heals the cracks and contrasts with the buff or gray montmorillonite of the matrix (Fig. 1 (*A*)). Slightly lower indices of refraction of the white montmorillonite in the veins suggest that its iron content is somewhat lower than that of the enclosing buff clay.

Several periods of clay migration are indicated by the intersection of these irregular veins, but it is difficult to determine the sequence of vein filling. However, in a specimen of clay composed of the closely related mineral beidellite from Twin Falls, Idaho, in the collection of C. S. Ross, at least four periods of fracture filling can be established. In Fig. 1 (*B*), photographed with nicols crossed, the veins are numbered in the order of their formation, which can be established by observing the cross cutting of early veins by late ones and the relation of veins to the fractures along which some but not others are offset. The mineral in the veins and in the matrix is beidellite and the only difference between the beidellite of the successive periods at Twin Falls is a slight difference in iron content. The prominence with which the veins stand out between crossed nicols results from the beidellite in the veins having most of its plates oriented parallel to the walls so that sections cut normal to the veins give nearly maximum birefringence and contrast with the somewhat random organization of the matrix.

In bentonites, which contain unbroken bubbles that were originally composed of volcanic glass but are now altered to montmorillonite, some movement of montmorillonite was necessary to fill the interiors of the bubbles with clay. When these bubbles were originally formed of volcanic glass, they contained gas; to give support to the walls and to prevent collapse of the structure under the weight of the overlying sediments, filling of the cavities with clay was essential. Many of these structures have been photographed by C. S. Ross (Fig. 2 (*A*)) and show plates of montmorillonite arranged perpendicular to the walls of the bubbles.[9] Since the interiors of the bubbles are now filled with clay and the walls appear unbroken, clay apparently

[9] C. S. Ross and E. V. Shannon, "Minerals of Bentonite and Related Clays and Their Physical Properties," *Jour. Amer. Ceram. Soc.*, **9** [2] 77–96 (1926).

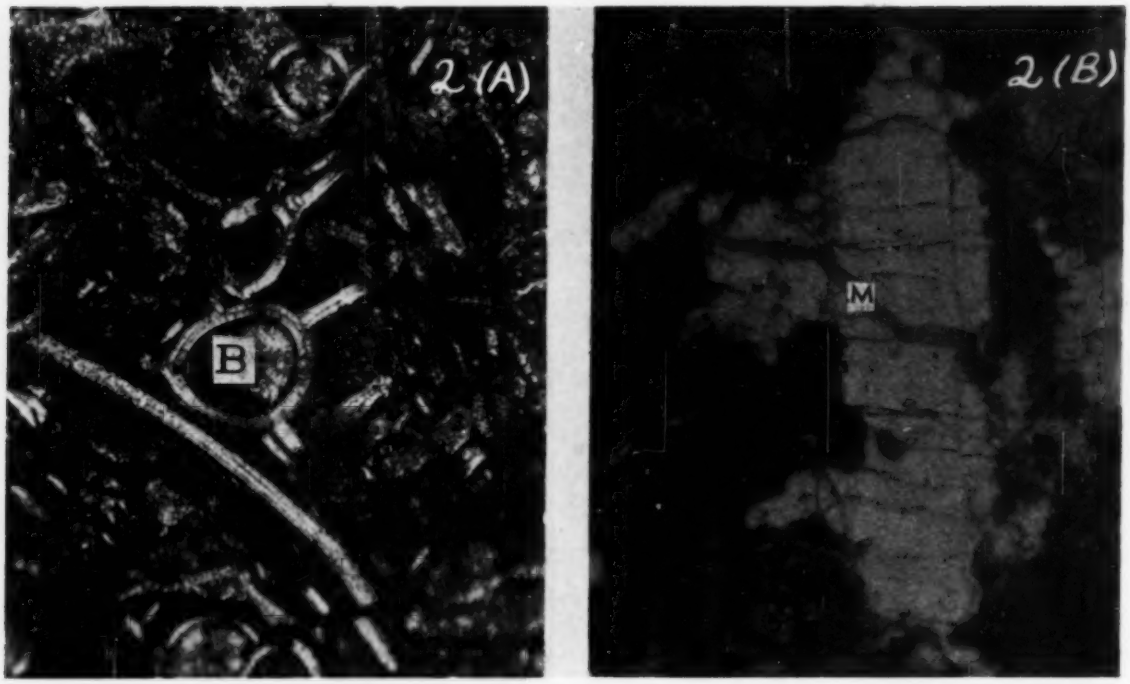

FIG. 2.—(*A*) Montmorillonite clay, filling bubble (*B*) originally of volcanic glass but now altered to montmorillonite in bentonite, San Diego County, Calif.; crossed nicols, ×150; (*B*) montmorillonite vein (*M*) cutting feldspar, Magnet Cove, Ark., ×150; both photographs by C. S. Ross.

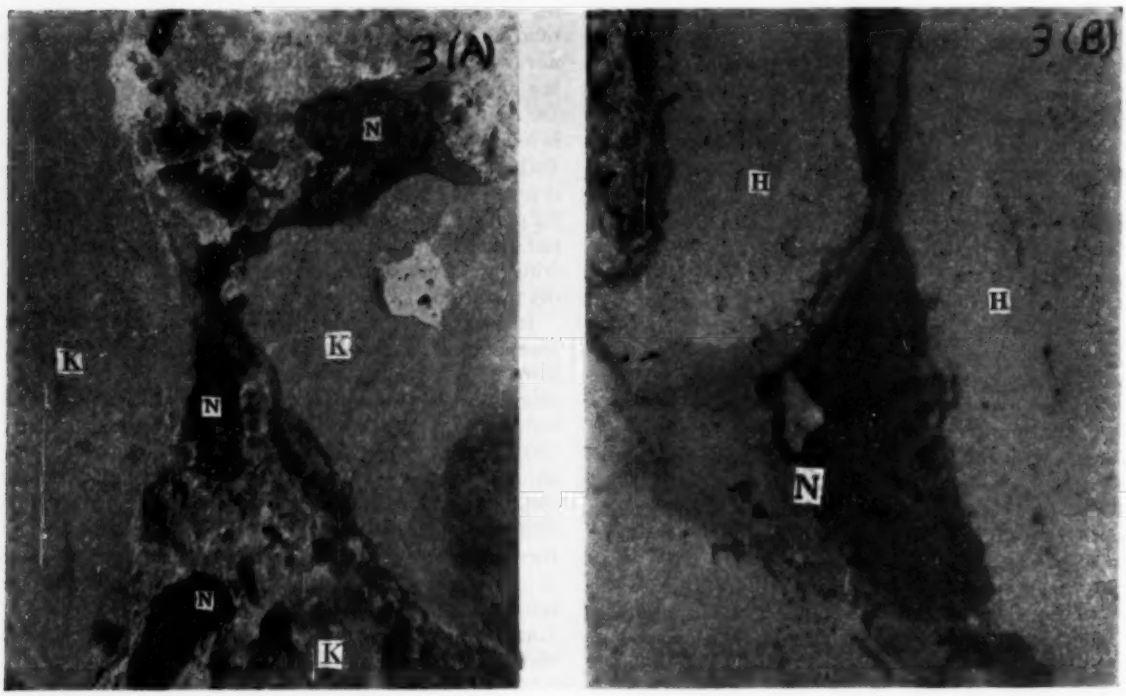

FIG. 3.—(*A*) Nontronite (*N*) filling fracture zone in kaolinite (*K*) of Tuscaloosa formation, Irwinton, Ga., ×150; (*B*) nontronite (*N*) along crack in halloysite vein (*H*), Eola Hills, Salem, Oreg., ×85.

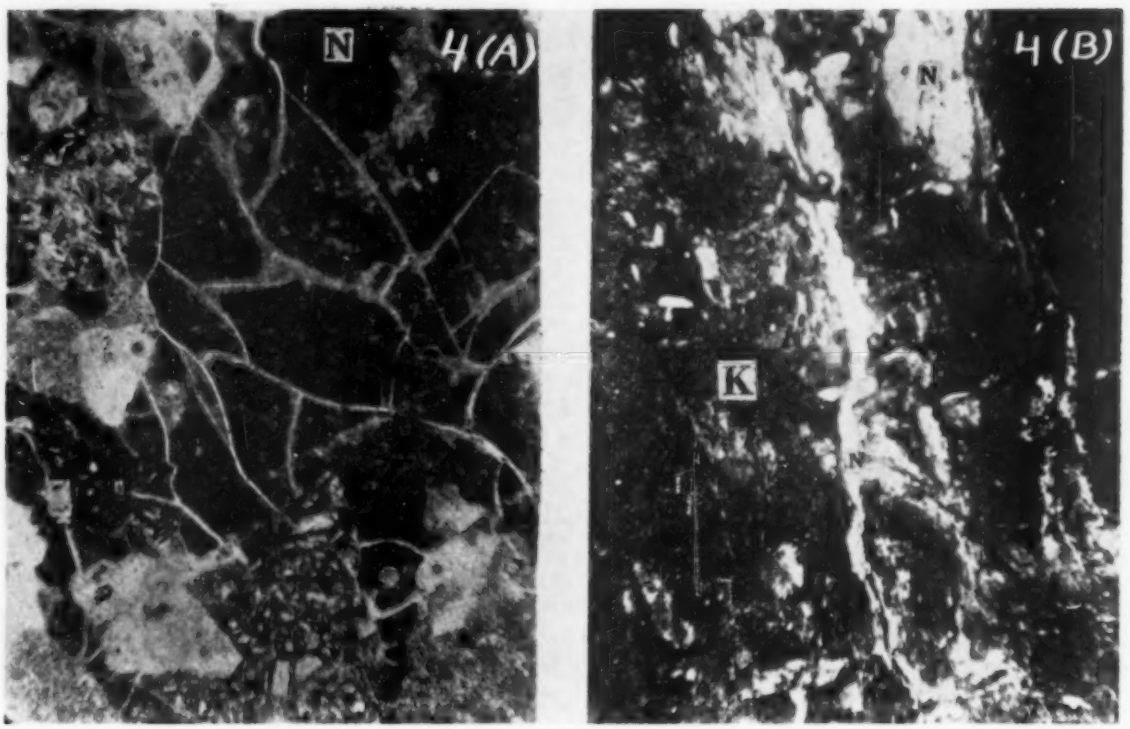

FIG. 4.—(*A*) Secondary nontronite along cracks in nontronite (black areas, *N*) cementing andesitic detritis, Molalla, Oreg., ×150; (*B*) nontronite (white, *N*) cutting kaolinite clay (dark, *K*); Excelsior clay deposit, near Spokane, Wash., ×150; crossed nicols.

migrated through the walls or through cracks in them to fill the interiors.

Numerous examples can be cited in which montmorillonite fills cracks in minerals or rocks that are too fresh or are otherwise unfavorably constituted to be the source material of the montmorillonite. In Fig. 2 (*B*), montmorillonite fills a crack in unaltered feldspar from Magnet Cove, Arkansas. The montmorillonite was derived from a source outside of the feldspar grain and has moved along cracks in the feldspar.

III. Migration of Nontronite and Halloysite

Nontronite is a member of the montmorillonite group in which ferric iron takes the place of alumina in the crystal structure. Its relatively high indices of refraction, its yellow-green color in hand specimen and its brown color in thin section resulting from heating it make possible the recognition of small areas of nontronite localized among other clay minerals. To the ceramist, the addition of appreciable amounts of secondary nontronite to a clay is of interest because the firing and other properties of the resulting clay mixture are modified proportionally.

At Irwinton, Georgia, nontronite fills fractures that form a zone in the sedimentary kaolin of the Tuscaloosa formation of Cretaceous age (Fig. 3 (*A*)) and line irregular cavities in kaolin of the "chimney rock" type. The relations indicate that the nontronite is secondary; it probably was carried downward from the clays of the Jackson group which once overlay the Tuscaloosa formation in this region.

A vein of white halloysite clay, ¾ in. thick, cuts the Stayton lavas in the Eola Hills, west of Salem, Oregon. Not only has halloysite moved along a fracture in the basalt at this locality but nontronite in turn has penetrated the halloysite along minor cracks (Fig. 3 (*B*)).

Green nontronite cements andesitic detritus occurring along the new Ostrander logging road, about three miles southeast of Molalla, Oregon (Fig. 4 (*A*)). The nontronite is cracked and pale-green nontronite fills the cracks. The relationship suggests that the cracks were formed when the clay dried and that the pale-green nontronite was deposited in the cracks when the clay was again saturated with water.

In the Excelsior clay deposit, seven miles southeast of Spokane, Washington, nontronite fills cracks formed in a kaolinitic clay derived from the weathering of basalt (Fig. 4 (*B*)). In one specimen of this clay, the nontronite veins are so narrow that they could easily be overlooked, but in other specimens they range up to 6 in. in width.

IV. Migration of Kaolinite

Although kaolinite may not migrate as easily as the minerals of the montmorillonite group, the selected examples which follow indicate that migration of kaolinite is not uncommon.

At the railroad cut near Vassar, Latah County, Idaho, a kaolinite vein cuts a residual clay derived from basalt in which lath-shaped feldspars have been altered to kaolinite (Fig. 5 (*A*)). The presence of a

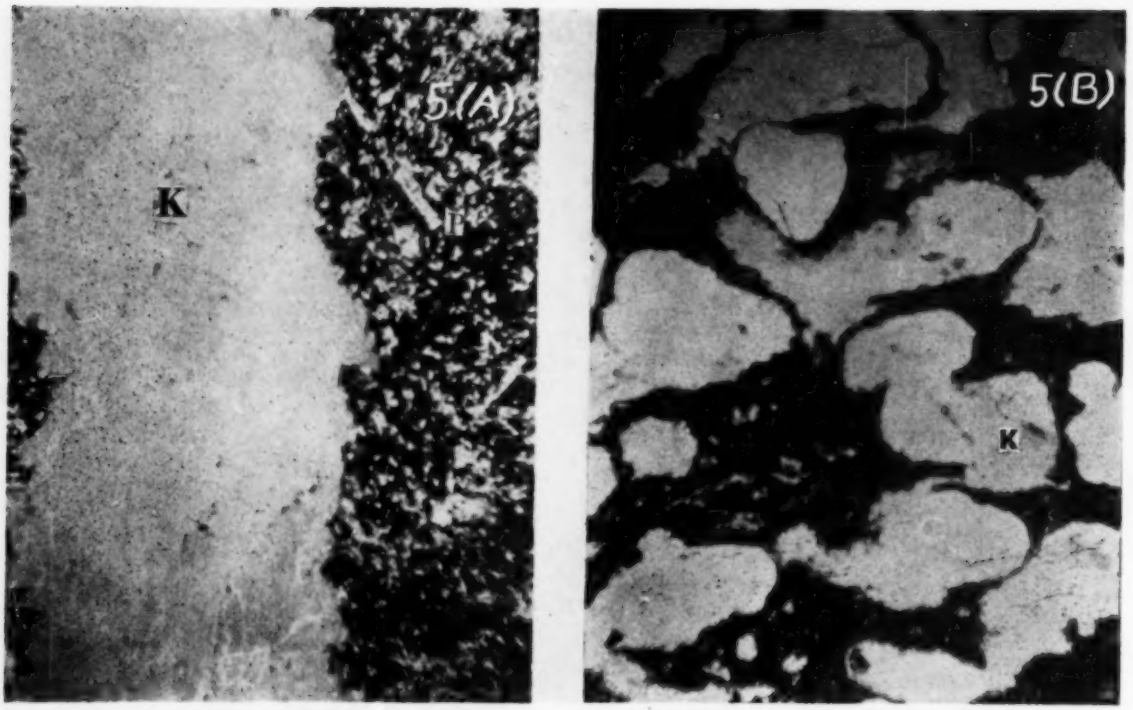

FIG. 5.—(*A*) Kaolinite vein (*K*) cutting residual clay formed from basalt with plagioclase (*P*), in basalt, altered to kaolinite retaining original shape, Vassar, Idaho, ×50; (*B*) kaolinite (*K*) filling vesicles in weathered basalt, Pullman, Wash., ×50.

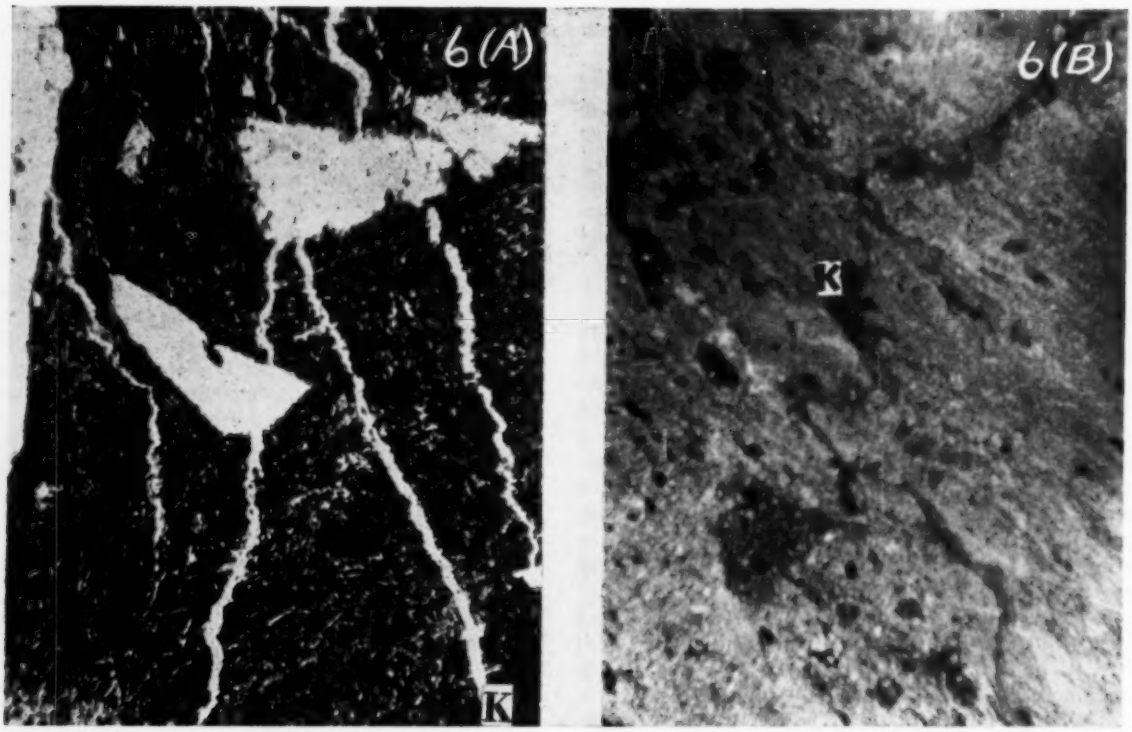

FIG. 6.—(*A*) Kaolinite veins (white, *K*) in altered porphyritic rock; plagioclase phenocrysts and tiny feldspars in groundmass altered to kaolinite, Mehama, Marion County, Oreg., ×150; (*B*) irregular vein of kaolinite (*K*), stained with organic matter, in flint clay; dark areas are kaolinite stained with organic matter, Olive Hill, Ky., ×85.

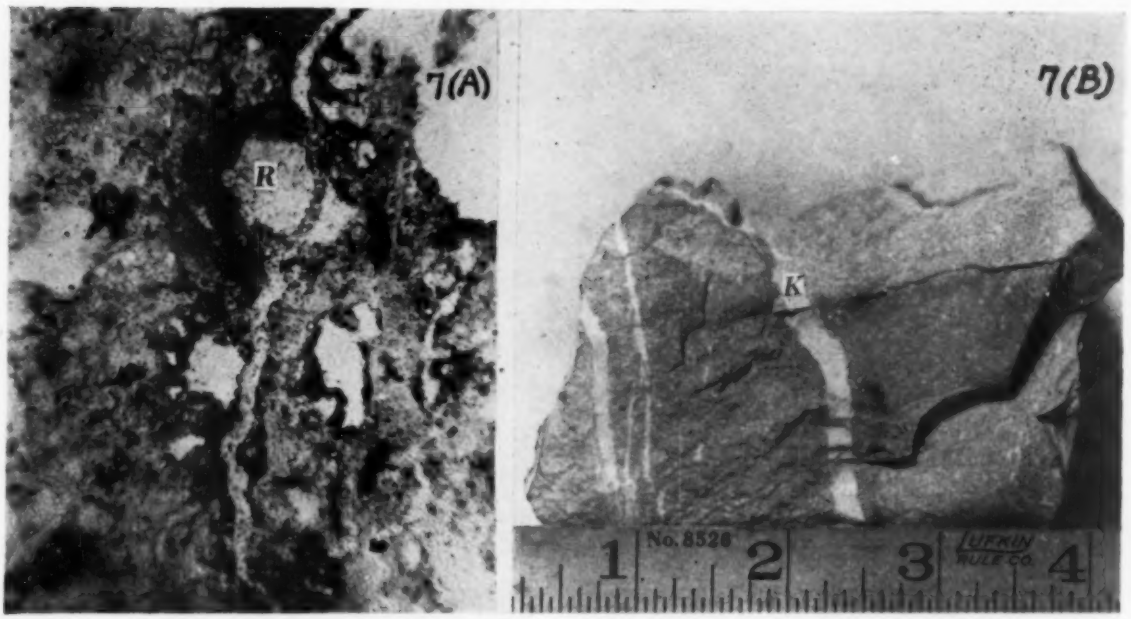

FIG. 7.—(*A*) Kaolinite and fine quartz in a vein cutting clay pellets and rock fragments (*R*) in water-laid breccia, Hobart Butte, Lane County, Oreg., ×150; (*B*) white veins of kaolinite (*K*) and fine quartz cutting light-colored refractory clay, Hobart Butte, Oreg.

few wisps of muscovite surrounded by the kaolinite in the vein and their absence in the altered basalt suggest that the kaolinite and muscovite in the vein migrated from one of the sedimentary clays that contain these minerals and are interbedded with the basalts of this region.

Along a road cut on highway 3, at the east edge of Pullman, Washington, the vesicles of a weathered basalt are filled with white kaolinite or halloysite (Fig. 5 (*B*)). Similar occurrences are present in the Excelsior clay deposit south of Spokane, Washington, where the residual clay contains plates of ilmenite partly altered to leucoxene, but no muscovite or quartz, whereas the white kaolinitic clay in the vesicles contains tiny muscovite flakes and quartz grains but no ilmenite. Muscovite and quartz are present in almost every sedimentary kaolinite clay in the region, and so it is reasonable to assume that the clay in the vesicles was derived from sedimentary clay. If this origin is correct, it brings forward an interesting problem of how the white clay containing quartz, muscovite, and kaolinite was transported through several inches of altered vesicular basalt. The vesicles do not join, nor do cracks appear to be present that could serve as channels along which the clay could be transported. The possibility remains that the residual clay once had sufficient permeability throughout the depth penetrated to allow ground water to carry white clay from the surface into the vesicles, but further alteration and compaction of the residual clay closed and obscured any visible channels.

In the Mehama district, Marion County, Oregon, porphyritic volcanic rocks have been altered to clay. The original feldspar phenocrysts are now kaolinite and veins of white kaolinite extend from one altered phenocryst to another, indicating a transfer of kaolinite from one decomposed feldspar to another (Fig. 6 (*A*)). The whiteness of the veins contrasts with the groundmass of this volcanic rock that now consists of altered tiny plagioclase feldspars in a matrix that is spotted with red iron oxides.

Refractory clays that are free from iron staining lack a color contrast that would aid in distinguishing early iron-stained kaolinite from later unstained clay minerals. Careful search, nevertheless, has revealed evidence of late movement of kaolinite at several localities, including the north-central Missouri fireclay district and the southeastern Missouri kaolin district. At Olive Hill, Kentucky, a white flint clay contains irregular veins of kaolinite stained brown with organic matter (Fig. 6 (*B*)). It is the organic matter in the veins that differentiates them from the inclosing white clay which composes the bulk of the specimen. The organic matter in the clay of the veins also gave it distinctive properties and aided in localizing it along the irregular cracks formed in the somewhat brittle clay. If sufficient clay from these microscopic veins could be separated and tested, its ceramic properties would probably approach more nearly those of a ball clay than those of the predominating clay of the matrix, which is free from organic matter. Where clay containing organic matter is carried downward from a higher to a lower bed, the properties of the clay of the lower bed may be modified accordingly.

V. Migration of Dickite and Hydrothermal Kaolinite

Dickite has the same chemical composition as

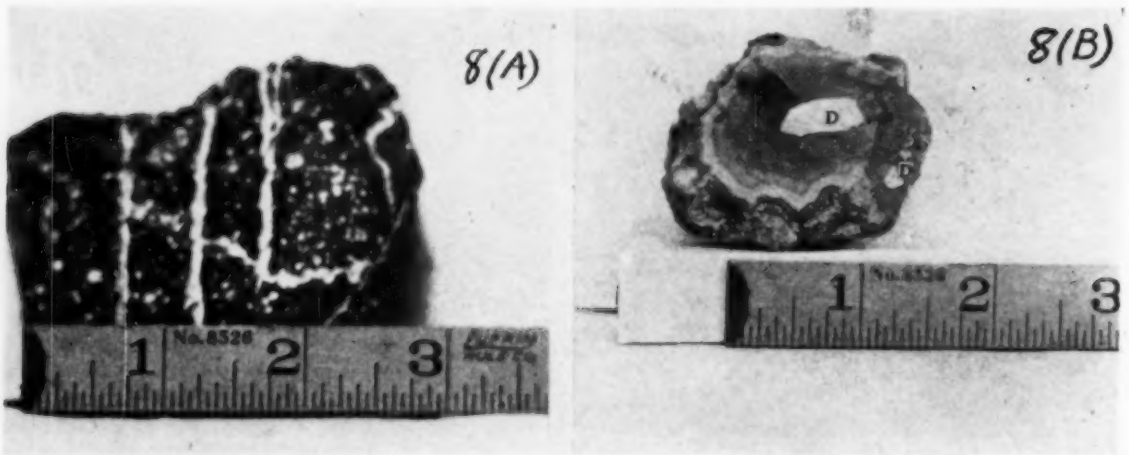

FIG. 8.—(*A*) Red iron-stained clay containing white clay pellets and cut by white veins of kaolinite and fine quartz, Hobart Butte, Oreg.; (*B*) geode of chalcedony and quartz enclosing white dickite (*D*) at center and lower right, St. Louis County, Mo.

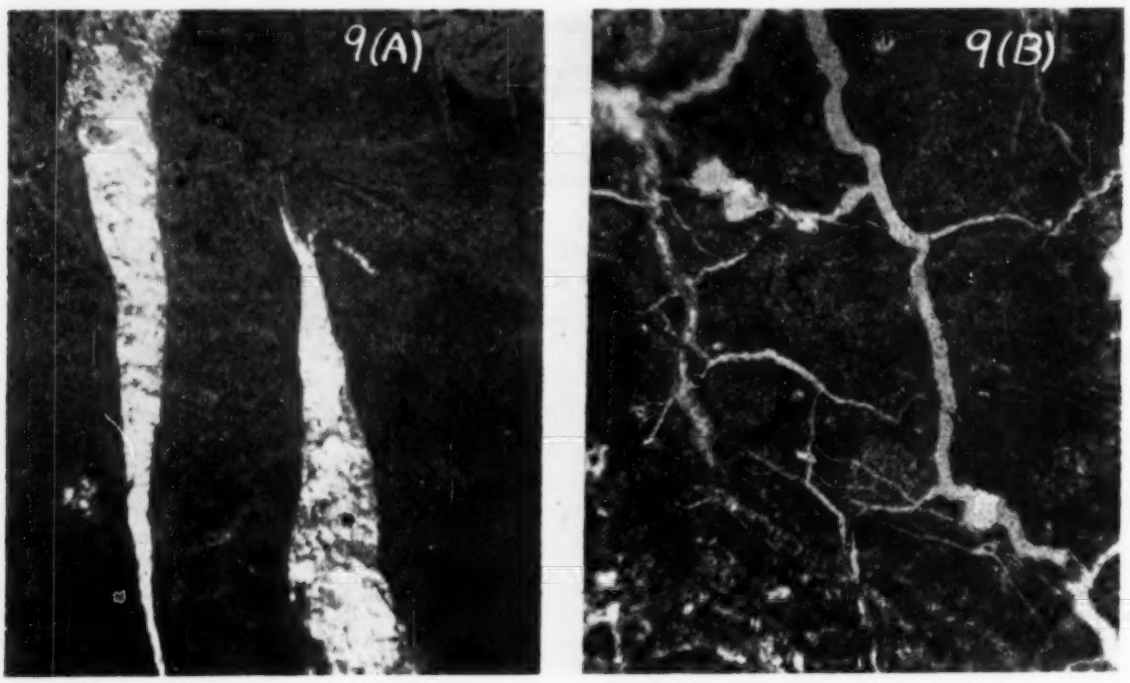

FIG. 9.—(*A*) Gibbsite veins (white) in a sedimentary kaolin, Wilkinson County, Ga., ×150; (*B*) gibbsite in irregular veins (white) cutting bauxite, Andersonville, Sumter County, Ga., ×150.

kaolinite but differs from it in optical properties and in its X-ray pattern.[10] It[11] has been prepared in the laboratory at a slightly higher temperature than that required to form kaolinite; the relations at some deposits indicate that it was deposited there by hydrothermal solutions.

At Hobart Butte, Lane County, Oregon, refractory clays containing pellet structures and organic matter were deposited by sedimentary processes.[12] Veins of white clay cut across the early minerals and rock structures (Fig. 7 (*A*) and (*B*)). In thin sections, the clay

[10] C. S. Ross and P. F. Kerr, "Kaolin Minerals," *Jour. Amer. Ceram. Soc.*, **12** [3] 151–60 (1929); also in *U. S. Geol. Survey Prof. Paper*, No. 165E, pp. 161–76 (1930).

[11] R. H. Ewell and Herbert Insley, "Hydrothermal Synthesis of Kaolinite, Dickite, Beidellite, and Nontronite," *Jour. Research. Nat. Bur. Standards*, **15** [2] 173–86 (1935); *Ceram. Abs.*, **14** [11] 290 (1935).

[12] V. T. Allen and R. L. Nichols, "Clay-Pellet Conglomerates at Hobart Butte, Lane County, Oregon," *Jour. Sediment. Petrol.*, **15** [1] 25–33 (1945).

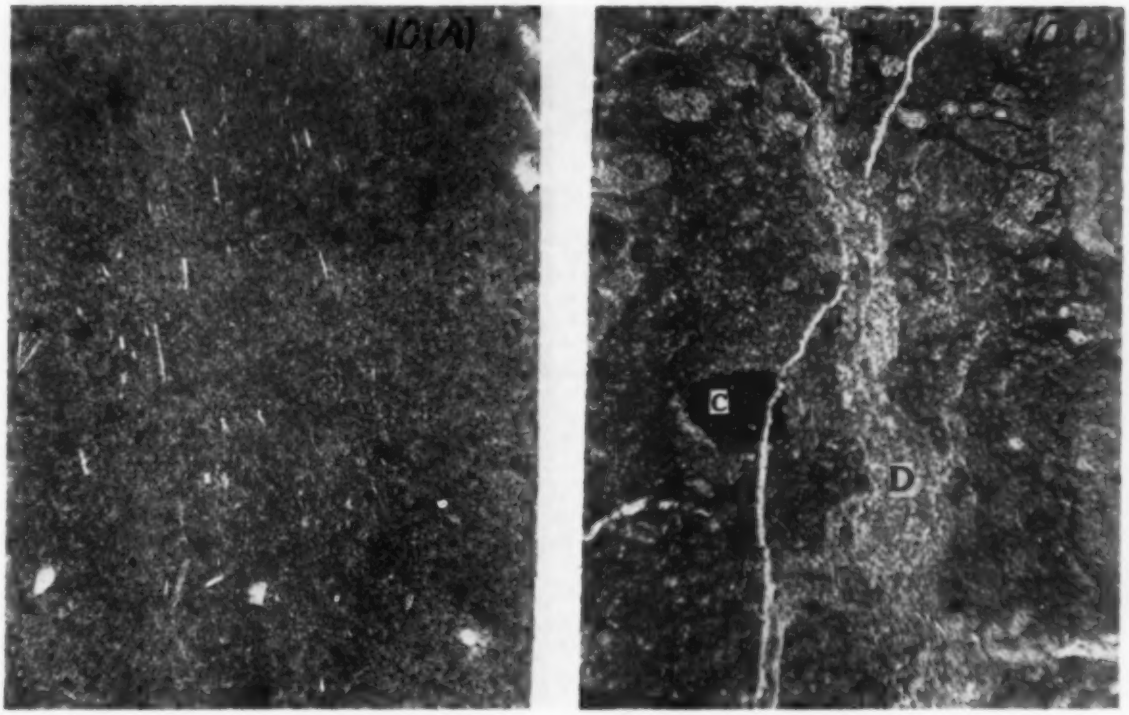

FIG. 10.—(*A*) Section of gibbsite tube with muscovite flakes arranged with long dimensions parallel to length of tube, Toomsboro, Ga., ×150; (*B*) diaspore filling veins and replacing structures in upper part of Cle Elum iron deposit, Kittitas County, Wash.; dark area (*C*) at left center is chromite cut by vein of chlorite, which is partly replaced by diaspore near lower fourth of photograph; this diaspore vein swings to right and joins main vertical diaspore vein (*D*); ovate areas were clay pellets of kaolinite and boehmite now partly or wholly replaced by diaspore, ×150.

mineral in the veins shows the mosaic intergrowth characteristic of the massive dickite from Chihuahua, Mexico, and Neurode, Silesia, and a few small grains have an extinction angle of about 16° that is indicative of dickite rather than kaolinite. All the clay samples, however, collected from veins at this locality, including one in which a few grains had an extinction angle of 16°, gave the X-ray pattern of kaolinite and quartz. This suggests that the amount of dickite present is too small to be recorded in the X-ray pattern of a mixture which is mainly kaolinite and that the temperature of the hydrothermal solutions may have been sufficiently high at first to allow some dickite to crystallize but was soon lowered to a stage where kaolinite crystallized. The realgar, stibnite, mansfieldite,[13] and scorodite closely associated with the kaolinite-dickite veins strongly suggest that the kaolin minerals in the veins were deposited by hydrothermal solutions. The quartz introduced with the kaolinite in the veins is so fine grained that it escapes detection with a microscope except in a few specimens in which quartz of microscopic size occurs as prismatic crystals with well-developed terminations. This finely divided quartz probably modified the ceramic properties of the light-colored clays, which are low in iron and refractory, more than did the kaolinite and dickite introduced in them by hydrothermal solutions (Fig. 7 (*B*)). In the Hobart Butte clays, high in iron like that illustrated in Fig. 8 (*A*), the effect was somewhat different. Here, the additions of low iron kaolinite and quartz improved the refractory properties of the resulting clay by decreasing proportionally the total iron.

[13] V. T. Allen and J. J. Fahey, "Mansfieldite, a New Arsenate, the Aluminum Analogue of Scorodite, Its Occurrence at Hobart Butte, Oregon," *Amer. Mineral.*, in press.

VI. Migration of Gibbsite

The presence of gibbsite, the trihydrate of alumina, in a clay is detrimental if the clay is to be used in the paper industry but it is advantageous if the clay is to be used in ceramics since gibbsite adds to the refractoriness of the clay. In Georgia, Alabama, Mississippi, and Arkansas, clays that contain gibbsite have been studied by the writer.

In Wilkinson County, Georgia, the Tuscaloosa formation of Cretaceous age contains lenses of sedimentary kaolin in which gibbsite fills secondary veins in a manner indicating that the gibbsite is being transferred from the matrix into fractures (Fig. 9 (*A*)).

In the Andersonville district, Sumter County, Georgia, bodies of pisolitic bauxite occur in kaolin lenses of Lower Eocene age. Pisolitic structure is developed in mixtures of fine-grained gibbsite and kaolinite. Crystalline gibbsite fills cracks that cut across the early structures of the bauxite (Fig. 9 (*B*)) and afford evidence of a secondary movement of gibbsite.

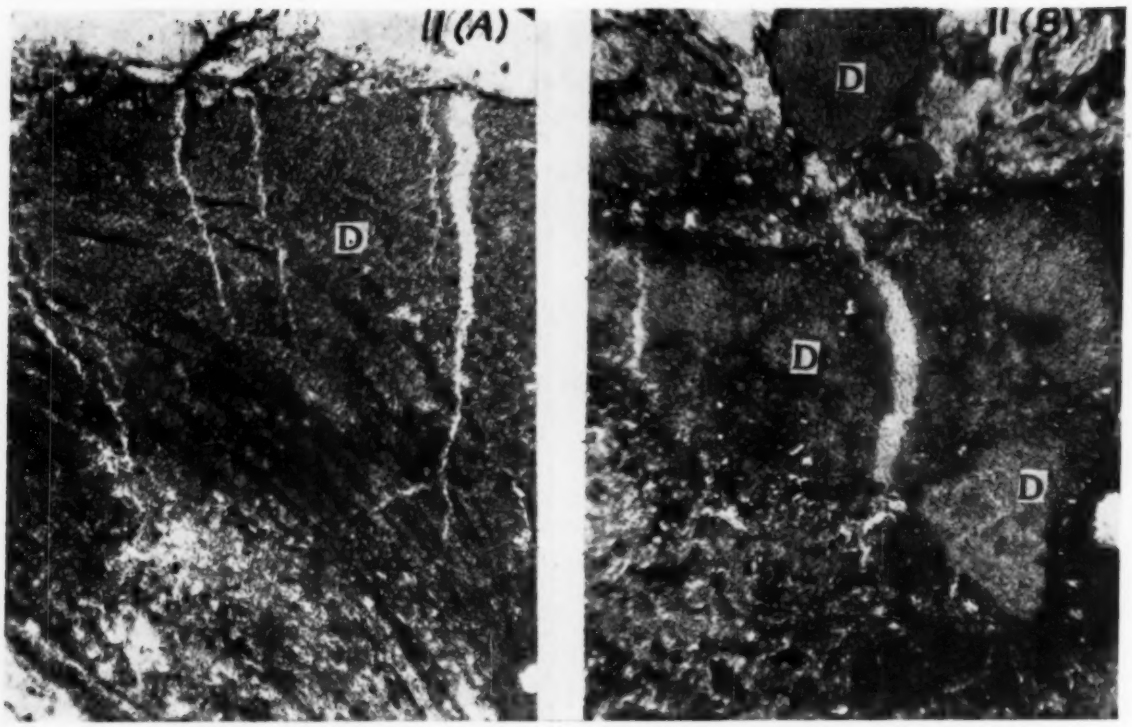

FIG. 11.—(*A*) Concentric structure in flint clay, originally kaolinite, replaced by fine-grained diaspore (*D*); white veins of second generation of coarse diaspore cut early diaspore; nodular clay, Clearfield County, Pa., ×260; (*B*) pellets of kaolinite are replaced by fine-grained diaspore (*D*); second generation diaspore (white) fills cracks cutting early diaspore, ×150; nodular clay, Clearfield County, Pa.

Near Toomsboro, Wilkinson County, Georgia, cylindrical tubes, 1/4 to 3/4 in. in diameter and 2 to 3 in. long, lie exposed on the surface detached from the clay. Optical, X-ray, and differential heating studies indicate that the tubes are composed of gibbsite with minor amounts of kaolinite, muscovite, and quartz. Thin sections, cut parallel to the long axes of the tubes, show residual areas of kaolinite and plates of muscovite arranged with their long dimension parallel to the length of the tubes (Fig. 10 (*A*)). A similar gibbsite tube but containing more quartz grains was found in place in clay pit No. 10, Dry Branch, Twiggs County, Georgia, where the relations in thin section indicate that gibbsite has replaced not only kaolinite but also quartz. These secondary concentrations of gibbsite in a tubelike form were developed by the movement of gibbsite which oriented the muscovite plates parallel to the length of the growing tube.

VII. Migration of Diaspore

The monohydrate of alumina, diaspore, occurs in well-defined veins in the upper part of the Cle Elum iron deposit, Kittitas County, Washington. It replaces chlorite that is present in veins cutting chromite and other early minerals and structures (Fig. 10 (*B*)). It also fills veins that give no evidence of having originally been occupied by chlorite, and it replaces early minerals and structures, including pellets that were originally kaolinite and boehmite.

The commercially important refractory nodular clays of Clearfield County, Pennsylvania, contain two generations of diaspore. The first generation is fine grained and replaces pellets and other structures of a flint clay that was originally composed of kaolinite. The second generation of diaspore is coarse grained and fills veins that cut across the early fine-grained diaspore (Fig. 11 (*A*) and (*B*)). Late movement of diaspore along open cracks is indicated at these localities.

VIII. Conclusions

Under favorable physical-chemical conditions, the migration of clay minerals and hydrous aluminum oxides takes place. Movement is facilitated by the presence of cracks or openings that serve as channels for ground water. Redistribution of these minerals along openings is aided by active circulation of water under conditions of good drainage and particularly where dispersing agents are present in the solutions so that dispersed systems of finely divided mineral particles may be transported. Under a combination of conditions that is seldom attained except at elevated temperatures, transfer of their constituents may take place as true solutions. Because the permeability of clay is slight and the number of secondary openings varies from one part of the deposit to another, these processes increase local inhomogeneities in the clay. The examples cited from widely separated localities indicate that some of the variation in the composition

of clay met by the clay producer is related to these processes. For each situation where contrasts in color, mineral composition, and structure make it possible to recognize the operation of these processes, there are probably several times that number which lack contrasting features and so escape notice. The processes are therefore more important than appears from these citations.

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CRAZING OF ENAMEL ON STOVE TOPS DUE TO HEATING IN SERVICE*

By Roger L. Fellows

Abstract

Examination of stove tops that had crazed in service show two types of crazing. One is in the form of a network of cracks which may be attributed to thermal shock, while the other is in the form of parallel lines and occurs in the normal use of the stove without any particular abuse. In studying the latter type of failure, localized heating of the stove top causes a cross-bend failure of the enamel due to flexing from the heat, and the higher the temperature the less is the amount of deflection required to cause failure. The temperature necessary for this condition is lower than 550°F. Sample plates subjected to various tests afforded a means of studying the different variables that produce crazing in stove tops.

From these tests, the following observations are made: (1) Stove tops that have tensile strains in the enamel due to fabrication or assembly are likely to craze in the areas of these stresses if they are heated; (2) the lower the coefficient of expansion of the covercoat enamel, the less likely is the enamel to craze; (3) the lighter the weight of application of cover coat, the greater is the resistance to crazing; (4) the heavier the gauge of metal the greater is the resistance to crazing; and (5) heating the enamel over a long period of time tends to induce crazing.

I. Introduction

Since the adoption of the one-piece porcelain enamel top for electric stoves, a small percentage of the stoves show cracking or crazing in the enamel around the burner openings and over the flanges. Some of these failures occur shortly after the stoves are placed in service and others, not until after two or three years of satisfactory service. The crazing usually occurs in more or less parallel lines, but in some cases the crazing is a network pattern of fine cracks. This crazing is not to be confused with hairlines which are lines or grooves in the enamel formed during the processing of the enamel part.

The object of this investigation was to obtain fundamental information on enamels that would have a bearing on the crazing, to develop a test for this type of crazing, and to study the effect of enamel composition and application on this defect.

II. Review of Literature

Crazing of enamel on cast iron[1] can be remedied by lowering the coefficient of expansion of the enamel or in some cases by changes in the design of the castings. Thermal-shock tests,[2] in which the enameled specimens have been heated and then quenched in water, have been used for testing crazing of enamels; this type of crazing usually occurs in the form of a network of fine cracks.

Schurecht,[3] in studying the crazing of glazes on wall tile, has found that crazing from thermal shock usually follows a hexagonal pattern, while crazing due to stresses occurs in more or less parallel lines. He has also found that glazes in high compression have better thermal-shock resistance than those in low compression.

Bell and Koenig[4] have found that there is a definite correlation between glaze stress, flexural strength, and thermal endurance of a glazed vitreous china body. In

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* Forty-Seventh Annual Program, 1945 (Enamel Division, No. 12); presented at the Chicago Local Section Meeting, April 7, 1945. Received April 26, 1945.

[1] H. F. Staley, "Cause and Control of Crazing of Enamels on Cast Iron," *Trans. Amer. Ceram. Soc.*, **14**, 516–45 (1912).

[2] (*a*) J. T. Robson, "Comparative Crazing and Chipping of Wet- and Dry-Process Cast-Iron Enamels," *Jour. Amer Ceram. Soc.*, **8** [7] 563–66 (1924).
(*b*) C. J. Kinzie, "Apparatus for Testing Enamels and Glazes for Resistance to Crazing under Thermal Shock," *ibid.*, **15** [2] 112–15 (1932).
(*c*) E. H. Shands, "Thermal-Shock Tests for Porcelain Enamels," *Amer. Enameler*, **5** [2] 8 (1932); Enamel Bibliography, 1944 ed., p. 247.

[3] (*a*) H. G. Schurecht and G. R. Pole, "Some Special Types of Crazing," *Jour. Amer. Ceram. Soc.*, **15** [7] 632–37 (1932).
(*b*) H. G. Schurecht, "Fitting Glazes to Ceramic Bodies," *ibid.*, **26** [3] 93–98 (1943).

[4] W. C. Bell and J. H. Koenig, "Effect of Glaze Film on Properties of a Vitreous Body," *ibid.*, **24** [11] 341–48 (1941).

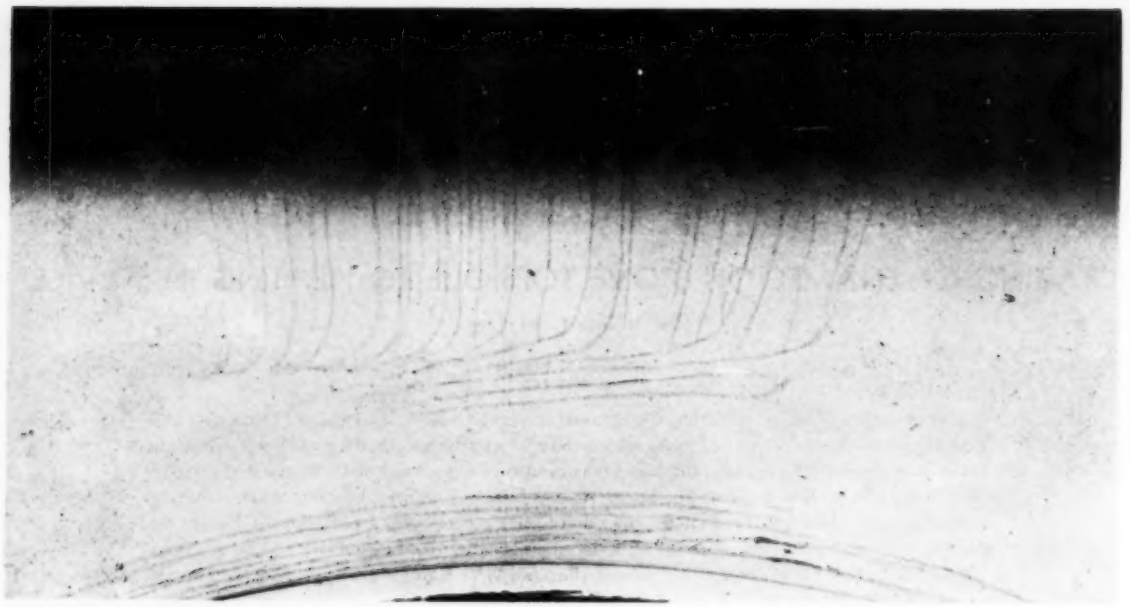

FIG. 1.—Craze lines around burner opening and over flange of electric stove top.

general, the glazes tested showed that the greater the glaze compression, the higher the flexural strength and the greater the thermal endurance.

III. Preliminary Tests

Examination of the craze lines on electric stove tops reveals that the majority are in the form of parallel lines; some, however, especially those close to the burners, are a network of cracks. This latter type of crazing is usually attributed to thermal shock and probably arises from wiping the hot stove with a wet rag or from water boiling over on the stove while in use. The parallel type of crazing as shown in Fig. 1 seems to have occurred while the stove was in normal use and without any particular abuse. This type of failure is the subject of the present investigation.

Preliminary tests revealed that if flat enameled panels were supported over an electric hot plate and heated uniformly from one side, crazing occurred when they reached 750° to 800°F., as measured with a surface pyrometer. The rupturing of the enamel was determined by rubbing the warm surface with the waxed side of carbon paper and subsequently removing the excess with a clean rag. Craze lines could also be produced by repeatedly heating similar enameled panels in a recirculating oven for 15 minutes at 800°F. In both of these cases, the crazing was irregular in design and followed no definite pattern. Specimens enameled at different weights of application ranging from 40 to 120 gm. per sq. ft. crazed at approximately the same temperature. Specimens that were enameled on both sides failed on both faces simultaneously.

These tests indicated that crazing results from a reversal in stresses from compressive to tensile which occurs during rapid heating of the enamel to approximately 800°F. The parallel type of crazing similar to that occurring on the stove tops was not reproduced in these preliminary tests of heating unstressed enameled iron specimens.

A pressed-steel electric stove top which had crazed badly in service was then tested to learn what treatment was necessary to produce crazing or to propagate that already present. The one-piece top with four burner holes on the left side had a back guard as an integral part. The top as removed from the main body of the stove was freed from the mounting strains that occurred in assembly, and in the following tests these strains need not be considered. The top was easily twisted, and it was noted that the craze lines which developed in service occurred in the section with the greatest freedom of movement.

The top was first cleaned thoroughly, and all of the crazed areas and scratches were marked with ink so that any new lines produced in the subsequent tests could be detected. The top was placed in a recirculating temperature-controlled oven at 250°F., held for 10 minutes, removed, allowed to cool to approximately 200°F., and examined for new craze lines or extension of the old ones. As no additional craze lines were noted, the test was repeated at 50°F. intervals up to 450°F. and then at temperatures of 550°, 650°, and 800°F. No additions or extensions of the original craze lines were brought out by these subsequent heatings.

To determine whether localized heating from the underside would cause crazing, the top was tested in the following manner:

A section of steel pipe, $6^1/_8$ in. outside diameter, $5^7/_{16}$ in. inside diameter, and $1^1/_2$ in. high, was centered on an 8-in. diameter, 2000-watt, 120-volt electric hot plate as shown in Fig. 2. A variable transformer and wattmeter were used to control heat input. After bringing the hot plate up to heat with a current input

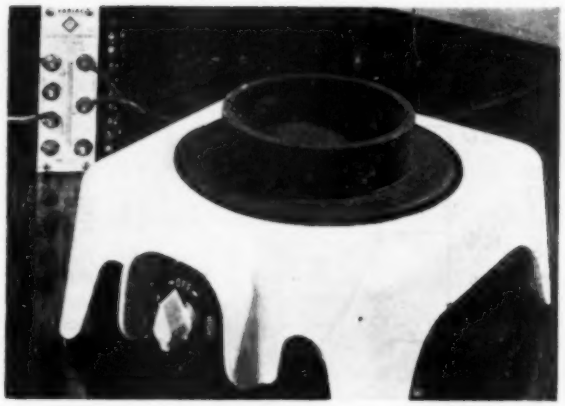

FIG. 2.—Photograph showing position of steel ring on hot plate.

of 1000 watts, the section of the stove top away from the burner openings was placed in contact with the ring. The corners were weighted down so as to hold the top firmly in place. While the top was heating up, cracking could be heard and the center of the heated area bulged upward. The temperature of the heated area as taken with a surface pyrometer was 520°F. at the hottest spot. After 15 minutes, the top was removed, cooled, and examined for crazing by rubbing with carbon paper. A number of parallel craze lines were produced which were similar in appearance to those formed in service.

A similar test was made on the area adjacent to that where the craze lines had been formed in service. The additional lines resulting were similar to the original and in most cases were extensions of them. Similar tests made at other locations on the stove top produced typical craze lines, the severity of which seemed to depend on the freedom of the part to bulge from heat; that is, the crazes were more readily produced in sections of the top that were weak in construction and easy to deform, such as those between the burner openings.

IV. Deflection Tests

The preliminary tests indicated that crazing in stove tops may result from heating in the range of 300° to 500°F. and frequently follows along lines of stress introduced in fabrication and assembly. The tests further showed that movement of the metal resulting from localized heating produced crazing and that failure does not necessarily occur at the point of maximum temperature. The cracking or crazing of the enamel appeared to be a flexure failure.[5] On the basis of this observation, further work was undertaken in order to study the effect of temperature, coefficient of expansion, and weight of application on the resistance of an enamel to failure under deflection.

[5] (*a*) R. R. Danielson and W. C. Lindemann, "Method for Testing Cross-Bending Strength of Enamels," *Jour. Amer. Ceram. Soc.*, **8** [12] 795–98 (1925).
(*b*) "Tentative Method for Determination of Resistance of Sheet-Steel Enamels to Deflection," *Bull. Amer. Ceram. Soc.*, **9** [9] 270 (1930); **7** [12] 360 (1928).

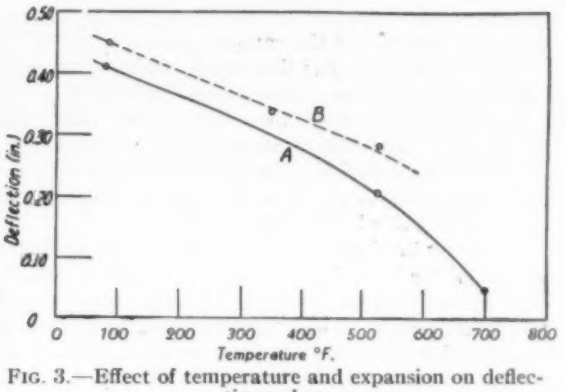

FIG. 3.—Effect of temperature and expansion on deflection value.

TABLE I

EFFECT OF TEMPERATURE AND EXPANSION ON DEFLECTION VALUE

Temp. (°F.)	Failure of specimens (in.) 1	2	3	4	5	Deflection value (in.)
Enamel A expansion (325 × 10^{-7} cm./cm./°C.)						
80	0.40	0.46	0.38	0.38	0.42	0.41
350	.30	.32	.28	.30	.30	.30
525	.24	.18	.22	.22	.20	.21
700	.04	.04	.04	.06	.06	.05
Enamel B expansion (291 × 10^{-7} cm./cm./°C.)						
80	.44	.42	.44	.46	.50	.45
350	.36	.34	.32	.34	.34	.34
525	.30	.26	.30	.28	.24	.28

The effect of temperature on the cross-bend strength was determined on a superopaque antimony-type white steel enamel A. The frit was milled with 7% clay, 2% opacifier, and $^1/_8$% sodium nitrite, and the enamel was applied at 60 gm. per sq. ft. on 20-gauge specimens, which were fired in a hanging position.

Five of the panels were tested at room temperature in the Lindemann-Danielson cross-bend machine as described in the Tentative Standard Deflection Test of the Enamel Division.[5,b] The specimens were placed in the apparatus, and the center roll was moved up one notch (0.02 in.) every 5 minutes until failure occurred. The average of the results on the five specimens was taken as the average deflection value of the enamel.

A second set of five plates were tested in a similar manner at 350°F. by conducting the deflection test in a temperature-controlled recirculating oven. The apparatus and plates were heated in the oven to the required temperature for one hour before placing the specimens in position. Care was required when placing specimens in the apparatus in order to obtain the zero reading. Since the samples were flatter at the higher temperature and warped on cooling, it was necessary to compensate for the cooling effect caused by opening the oven door. By starting the test with the specimens about $^1/_4$ in. from making contact with the upper roll and by moving the center roll up a notch at a time, the point of contact could be accurately determined for each specimen as the test progressed. After holding at the desired temperature for 5 minutes, the plates were tested for

crazing. The roll was then moved another notch, the door was closed, and the samples were allowed to remain for 5 minutes after the oven returned to temperature. Similar tests were made at 525° and 700°F.

Another series of tests were made at 80°, 350°, and 525°F. on an antimony white enamel B having a lower coefficient of expansion. The results obtained from the tests on enamels A and B at the various temperatures are listed in Table I and plotted in Fig. 3.

Five cross-bend specimens of enamel A were prepared at each of the following weights of application: 40, 60, 80, 100, and 120 gm. per sq. ft. Five specimens of each weight of application were tested simultaneously in the cross-bend machine at room temperature, and the results are shown in Table II and plotted in Fig. 4.

TABLE II

EFFECT OF WEIGHT OF APPLICATION OF COVER COAT ON DEFLECTION VALUE

Application wt. (gm./ sq. ft.)	Total enamel thickness (in.)	Failure of specimens (in.)					Avg. deflection value (in.)
		1	2	3	4	5	
40	0.010	0.54	0.56	0.58	0.50	0.56	0.55
60	.014	.40	.40	.42	.36	.44	.40
80	.018	.30	.36	.34	.38	.30	.34
100	.022	.26	.28	.26	.28	.26	.27
120	.026	.26	.26	.24	.26	.24	.25

V. Thermal Failure of Prestressed Specimens

Since the tops were mounted onto the main body of the stove in such a way as to cause localized mechanical stress in the enamel, an effort was made to ascertain the effect of this prestressing on thermal failure. Five cross-bend strips prepared with enamel A at 60 gm. per sq. ft. were selected for uniformity so that when they were placed in the cross-bend machine, the rolls contacted all of the specimens at the same time. All of the specimens were then bent 0.20 in. at room temperature. The apparatus containing these specimens was heated in a recirculating oven to 150°F. for an hour to allow the apparatus to reach equilibrium. After examining for cracks or crazing while in the oven, the temperature was raised 50° and held for 15 minutes. The samples were re-examined, and the test was repeated at 50° intervals to 800°F. Five additional samples of similar nature were placed loosely in the oven and run simultaneously with the stressed specimens.

Of the five stressed specimens, one failed at 450°F., one at 500°F., and three at 550°F. The average of the temperatures of failure of the stressed specimens was 520°F. None of the unstressed specimens which had been placed loosely in the oven displayed cracking or crazing.

Five additional specimens of enamel A prepared as described were selected and bent 0.20 in. in the cross-bend machine as before. The apparatus containing the specimens was heated in the recirculating oven at 300°F. for 425 hours and periodically tested for cracks. Since no cracking occurred, the test was continued at 400°F. After 40 hours, all of the specimens showed failure.

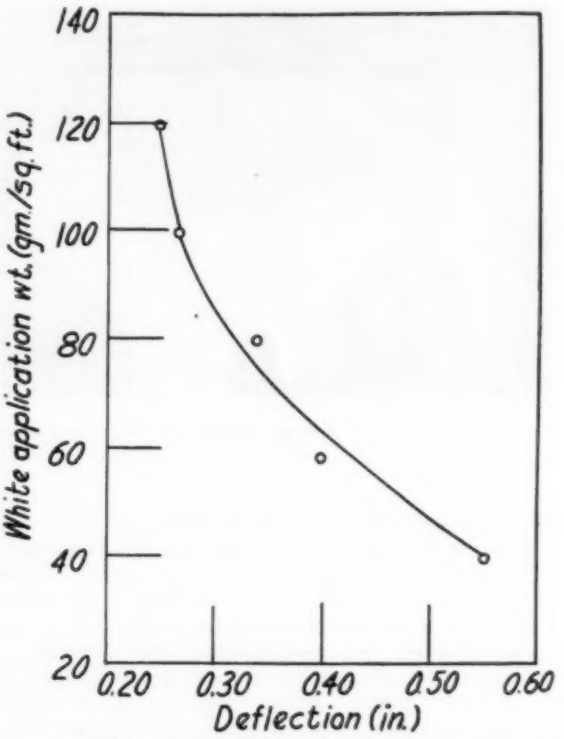

FIG. 4.—Effect of weight of application of cover coat on deflection value.

VI. Expansiometer Tests

The deflection tests in Table I show that only a small amount of deflection or force is necessary to cause the enamel to fail when the temperature of the specimen is at 700°F., which indicates that the compressive stresses are relieved at approximately this temperature. The deflection test, however, does not bring out the point at which the enamel changes from compression to tension on heating. Furthermore, the apparatus does not lend itself to ease of operation at the elevated temperatures. Because of this, the expansiometer test apparatus described by Rosenberg and Langerman[6] was adopted in a series of experiments.

Split-ring expansiometer specimens were enameled on both sides with ground coat and on the outside with white. Three grams of white enamel (50 gm. per sq. ft.) were applied to the specimens, dried, and then fired with one edge resting on the burning tool. Specimens were prepared from enamel A and from enamel B in this manner. The weight required to just open the ring was determined on one specimen at room temperature. The sample was then heated slowly in an oven to 500°F. After cooling to room temperature, the weight required to open the ring was remeasured. This process was repeated for three additional cycles at 700°F. The results on the two enamels so tested are shown in Table III.

[6] J. E. Rosenberg and A. Langerman, "Integral Expansion of Vitreous Enamels Between Softening Point and Room Temperature," *Jour. Amer. Ceram. Soc.*, **20** [7] 236–44 (1937)

During the heating cycles, the rings were carefully observed and the temperature of the oven was noted when the rings started to open. The results of these observations are listed in Table IV.

TABLE III

WEIGHT REQUIRED TO OPEN EXPANSIOMETER RINGS

Cubical coefficient of expansion	Enamel A (gm.) 325×10^{-7} cm./cm./°C.	Enamel B (gm.) 291×10^{-7} cm./cm./°C.
After enameling	360	540
After heating to 500°F.	360	540
After reheating to 700°F.	200	410
1st reheating to 700°F.	200	410
2d reheating to 700°F.	200	410

TABLE IV

TEMPERATURE REQUIRED TO OPEN EXPANSIOMETER RINGS

	Enamel A (°F.)	Enamel B (°F.)
Heating to 700°F.	625	675
1st reheating to 700°F.	450	600
2d reheating to 700°F.	450	600

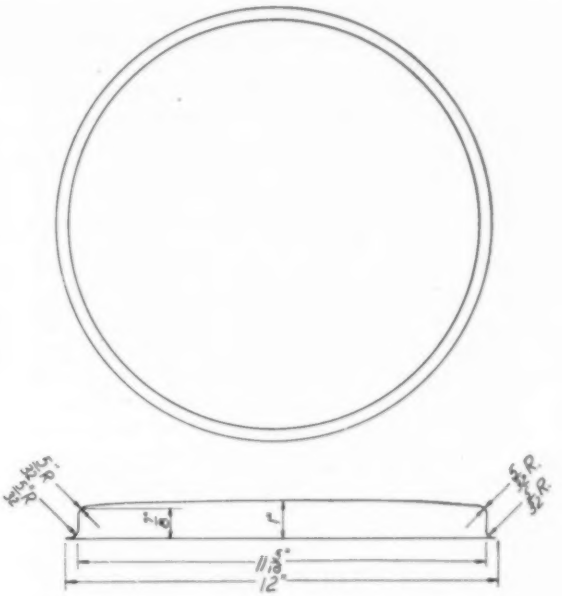

FIG. 5.—Rimbound test specimen.

VII. Rimbound Test Specimens

In the study of the problem thus far, some fundamental knowledge of the conditions that will affect the crazing of the enamel was obtained. In order to simulate more closely the condition of a stove top in use, rimbound specimens were made (see Fig. 5). Experiments were then conducted to determine the effect of weight of application, the coefficient of expansion, and the thickness of metal on the crazing tendency of the cover-coat enamel.

The rimbound panels were enameled with ground coat on both sides and with cover-coat enamel A on the outside of each of the following weights of application: 40, 50, 60, 80, and 120 gm. per sq. ft. The samples were supported on the flanges during firing.

Starting at room temperature, a specimen was heated over the hot plate illustrated in Fig. 2. Before heating, the initial flatness of the panel was determined with a dial-indicating arrangement as shown in Fig. 6. By means of this apparatus, readings were taken at 1-in. intervals and in two directions at right angles to each other with the center of the plate as the starting point. After making the traverses at room temperature, the hot plate which was controlled by a variable transformer was set at 100 watts and held for one hour. At the end of this period, the dial reading at the center of the panel was noted and the maximum surface temperature was recorded by means of a surface pyrometer. The enamel was then tested for crazing by rubbing carbon paper over the surface. The test was repeated at 100-watt intervals until the panel crazed, whereupon dial-indicating traverses were made at the initial points on the panel. Typical test data secured on one specimen are shown in Tables V and VI.

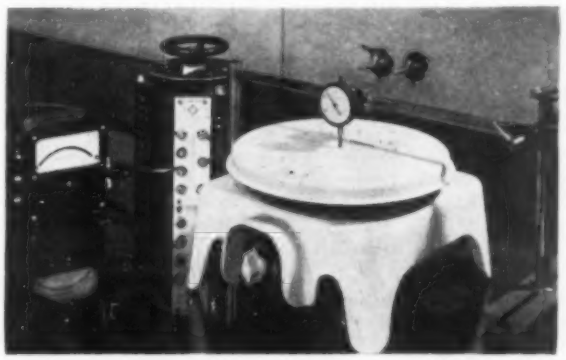

FIG. 6.—Dial indicator gauge in place on panel over ring on hot plate with variable transformer and wattmeter.

TABLE V

CENTER DEFLECTIONS AND SURFACE TEMPERATURES OF RIMBOUND SPECIMEN

Watts	Application wt. at 40 gm./sq. ft. Center deflection (in.)	Surface temp. (°F.)
100	+0.015	130
200	.036	200
300	.051	260
400	.064	310
500	.074	370
600	.082	410
700	.087	440
800	.095	480 (failed)

The distortion of the panel was determined from the data by taking the difference between the deflection at the center and 5 in. away in each direction. The

TABLE VI

FLATNESS TRAVERSES ON RIMBOUND SPECIMEN

Application wt. at 40 gm./sq. ft.

In.	North to south traverse (in.) Room temp.	480°F. (failure)	Deflection	West to east traverse (in.) Room temp.	480°F. (failure)	Deflection
5	−0.072	−0.140	−0.068	−0.054	−0.077	−0.023
4	− .047	− .081	− .034	− .038	− .030	+ .008
3	− .030	− .014	+ .016	− .022	− .020	+ .042
2	− .017	+ .041	+ .058	− .010	− .065	+ .075
1	− .008	+ .077	+ .085	− .005	− .089	+ .094
Center	0	+ .095	+ .095	0	+ .095	+ .095
1	+ .007	+ .097	+ .090	+ .004	+ .085	+ .081
2	+ .011	+ .079	+ .068	+ .006	+ .054	+ .048
3	+ .011	+ .041	+ .030	+ .006	+ .005	− .001
4	+ .005	− .015	− .020	+ .003	− .058	− .061
5	− .010	− .061	− .051	− .003	− .106	− .103

TABLE VII

EFFECT OF WEIGHT OF APPLICATION ON CRAZING OF RIMBOUND SPECIMENS

Application wt. of cover coat (gm./sq. ft.)	Total enamel thickness (in.)	Watts at failure	Temp. at failure (°F.)	Total center deflection (in.)	Max. deflection at failure (in.)
40	0.0105	800	480	0.095	0.198
50	.0125	800	470	.085	.174
60	.0140	800	470	.088	.160
80	.0175	500	350	.072	.134
120	.026	500	360	.073	.118

TABLE VIII

EFFECT OF METAL GAUGE ON CRAZING OF RIMBOUND SPECIMENS

Metal gauge	Watts at failure	Temp. at failure (°F.)	Total center deflection (in.)	Max. deflection at failure (in.)
20	800	470	0.088	0.160
24	800	470	.090	.192
26	600	420	.080	.156

highest value was taken as the maximum deflection of the panel in inches; an example follows.

Center to		In.
North	0.095 − (−0.068)	= 0.163
South	.095 − (− .051)	= 0.146
West	.095 − (− .023)	= 0.118
East	.095 − (− .103)	= 0.108

The largest of the four values is shown to be 0.163 in., which is therefore the maximum deflection at failure. Similar data were secured on the other panels enameled at different weights of application. This information is shown in Table VII and plotted in Fig. 7. The craze lines usually occurred in the areas of maximum deflection.

The effect of coefficient of expansion on the crazing tendency of rimbound panels was studied at one weight of application by applying enamel B to a panel at 60 gm. per sq. ft. and testing as previously described. Failure of this panel occurred at 900 watts or 510°F. as compared with 800 watts or 470°F. for enamel A under similar conditions.

Further studies on rimbound specimens were made on metal of different gauges. The initial studies at the different weights of application and expansions had been made with 20-gauge steel panels. Additional panels prepared from steel of 24 and 26 gauge were enameled at 60 gm. per sq. ft. with enamel A and tested as outlined in the preceding experiment. The results of these tests are compared in Table VIII.

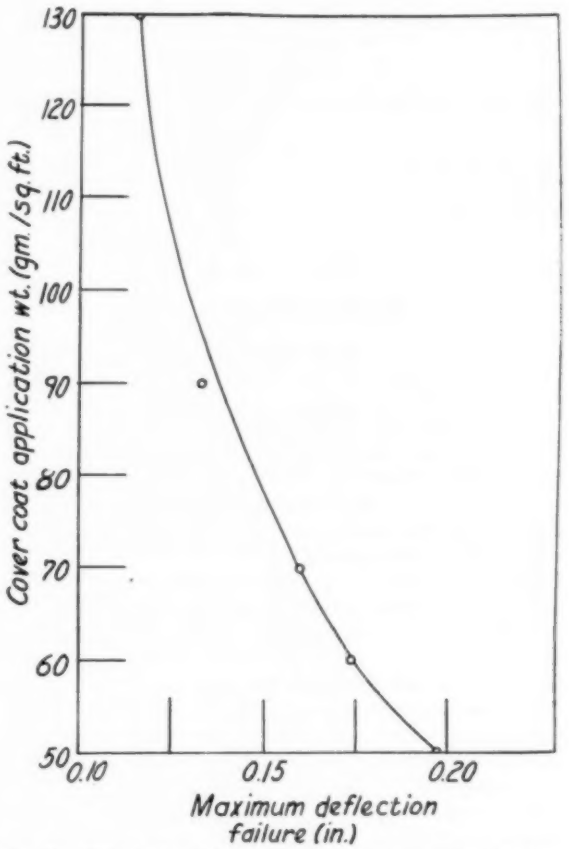

FIG. 7.—Effect of weight of application on crazing of rimbound specimens.

VIII. Discussion of Results

Localized rapid heating of stove tops produces crazing of the enamel due to flexure at relatively low temperatures.

Enamel A applied at 60 gm. per sq. ft. shows a decrease in deflection value from 0.41 in. at room temperature to 0.05 in. at 700°F. when tested in a cross-bend machine. The resistance of enamel A to cross-bend failure at 525°F. is approximately one half that at room temperature, which shows that enamel requires less bending to cause failure when heated.

When the cubical coefficient of expansion is decreased from 325 to 291, an increase in deflection of 0.04 in. results. The lower-expansion enamel shows a correspondingly higher resistance to deflection failure over the entire temperature testing range. As a result, the higher-expansion enamel cannot withstand as high a temperature as the lower-expansion type by 100°F. in order to have comparable deflection resistance.

The deflection tests further reveal that the deflection value decreases from 0.55 to 0.25 in. when the weight of application is increased from 40 up to 120 gm. per sq. ft. The resistance of enamel to cross-bend failure is as materially affected by decreasing weights of application as lowering of the expansion.

Good correlation of deflection test results was secured from the prestress experiment. When panels of enamel A were flexed 0.20 in. at room temperature and heated while under tension, failure occurred at a temperature (520°F.) corresponding to that on panels heated with increasing increments of stress and temperature (Fig. 3). Panels stressed in a similar manner failed when heated for a long period of time (440 hours) at a maximum of 400°F.

Expansiometer tests showed that more force (540 gm.) is required to open the ring on a lower-expansion enamel than that on a higher-expansion type (360 gm.). On a comparable basis, temperatures of 625° on the high-expansion and 675°F. on the low-expansion enamels were necessary to open the rings and they are, therefore, the temperatures at which the enamel stresses change from compression to tension. Repeated heatings in this test above these critical temperatures caused a lowering of the compressive stresses of both enamels (see Tables III and IV).

The deleterious effects of increasing weights of application on the crazing resistance of an enamel are substantiated by the tests on rimbound specimens. In Table VII, it may be seen that as the weight of application was increased from 40 up to 120 gm. per sq. ft., a corresponding decrease from 0.198 to 0.118 in. in the maximum deflection necessary for failure results. The amount of heat input necessary for failure also showed a decrease in the same test, which is equivalent to a lower temperature of failure. An exploratory test of very light application utilizing 0.008 in. of a special cover enamel applied directly on iron augmented the data herein reported. This rimbound specimen required 1000 watts (520°F.) to produce failure. The effect of eliminating the ground coat was determined when this latter enamel was applied at 0.014 in. and found to fail at 800 watts, which corresponds with previously reported results on test enamel A.

The use of lower-expansion enamel on rimbound specimens increased the crazing resistance and thereby confirmed the work of previous investigation as well as other data herein reported.

Due to the inability to secure specimens of heavier gauges, the results of this phase of the investigation are not as comprehensive or conclusive as desired. The trend of decreased crazing resistance with decreasing metal thickness, however, is indicated, and additional work should be done with heavier gauge material.

IX. Summary

Localized heating of the stove top causes a cross-bend failure of the enamel due to flexing from the heat, and the higher the temperature, the less the deflection required to cause failure. The temperature necessary for this condition occurs below 550°F. Sample plates subjected to various tests afforded a means to study the different variables producing crazing in stove tops. The conclusions and observations of this study are given in the abstract of the paper.

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METHOD FOR MEASURING FINING TIME OF GLASS*

By A. K. Lyle

Abstract

A method is described for measuring the fining time of glass, and a formula is given for using the data obtained to estimate the tonnage-temperature schedules of continuous tank furnaces. Measurement of fining time at two and preferably three temperatures, in the range 1400° to 1500°C., is required to establish the fining characteristics of a glass. Data from fining-time measurements of four glasses show that fining is largely independent of the viscosity and probably also of the surface tension of glass.

The use of fining-time measurements in accounting for differences in the melting efficiencies of tank furnaces and the value of systematic studies, involving fining agents and variations in composition, are suggested.

I. Introduction

Glass is produced from a mixture of crystalline materials by the two related processes of melting and fining. Melting is recognized by the formation of a liquid phase and is complete when all crystalline material has disappeared from the melt. Fining is the process by which gases, released by reactions among the batch materials, are eliminated from the molten glass; fining is complete when the melt no longer contains bubbles. The two processes operate concurrently, but fining always continues for some time after melting has ceased. Fining is, therefore, the limiting process in the making of glass.

Many writers have assumed that fining is closely related to viscosity. Morey,[1] in summarizing the importance of the viscosity of glass, states, "The rate of melting of glass is chiefly determined by the rate of removal of bubbles which, in turn, is largely dependent on the viscosity." Similar statements are made by Scholes[2] and by Hodkin and Cousen.[3]

Day,[4] however, using a truly quantitative method for determining the fining rate of glass, was unable to recognize any simple relation between the time required to eliminate bubbles and the viscosity of the glass. Unfortunately, the experiments reported by Day were preliminary in character, and details of the work were not published.

II. Purpose and Outline of Method

The purpose of this paper is to describe a method for determining the fining time of glass and to demonstrate its usefulness in estimating the fining rates of glasses melted in continuous furnaces. Data are presented to show that the rate of fining is largely independent of viscosity and of surface tension.

Under carefully controlled conditions, a small charge of pulverized batch is added to a platinum crucible containing a relatively large amount of previously melted and fined glass. At the end of a predetermined interval of time, a sample is poured from the crucible. When annealed and cooled, the sample is examined for bubbles. The fining time is noted as the minimum time required to produce a sample substantially free of bubbles as determined by a series of tests.

III. Equipment

The diagram of the furnace is shown in Fig. 1. The top and bottom insulation is 2.5 in. of B & W K-33 insulating firebrick. The side insulation is 4.5 in. of B & W K-33 and 2.5 in. of Armstrong insulating firebrick. The heated chamber is 9 in. high and 4 in. minimum diameter. Recesses, shaped as shown in the drawing, accommodate four Globar heating elements, which extend vertically through the furnace and are supported by insulated brackets. The pier for supporting the crucible is a cylindrical piece of B & W K-30 fashioned from a brick cut lengthwise and drilled to receive a thermocouple. The pier is held in an adjustable clamp fastened to a counterbalanced arm, which is movable vertically and at its lowest position can be swung clear of the furnace. Figure 2 shows the furnace and the pier with crucible in the lowered position.

The Globar elements are 18 in. long by 3/4 in. diameter and are designated 18 by 10 by 3/4 in., 46 v., 52 amp. The active part of the elements is confined to the central 10-in. portion, the 4 in. at each end being of low resistance and coated with aluminum. Connection to the power line is by means of spring clips lined with aluminum foil. Safety guards, not shown in Fig. 1, cover the protruding ends of the Globars.

The wiring diagram of the power and control circuits is shown in Fig. 3. Power is supplied by a 220-volt line through the transformer which is tapped for a range of 35 to 90 volts in five-volt steps. The heating elements are connected in parallel. As the resistance of the Globars increases with continued use, the voltage is increased so as to maintain a power input of about 4 kva. for temperatures from 1400° to 1500°C.

* Forty-Seventh Annual Program, The American Ceramic Society, 1945 (Glass Division, No. 5). Received May 9, 1945.

[1] G. W. Morey, Properties of Glass, p. 135. Amer. Chem. Monograph Series, Reinhold Publishing Corp., N. Y., 1938. 561 pp.; *Ceram. Abs.*, **18** [2] 48–49 (1939).

[2] S. R. Scholes, Modern Glass Practice, p. 150. Industrial Publications, Inc., Chicago, Ill., revised ed., 1941, reviewed in *Ceram. Abs.*, **20** [7] 168 (1941).

[3] F. W. Hodkin and A. Cousen, Textbook of Glass Technology, p. 43. Constable & Co., London, 1925; N. Y. ed. reviewed in *Ceram. Abs.*, **4** [8] 235–36 (1925).

[4] R. K. Day, unpublished report, abstracted in *Glass Ind.*, **12** [3] 47 (1931).

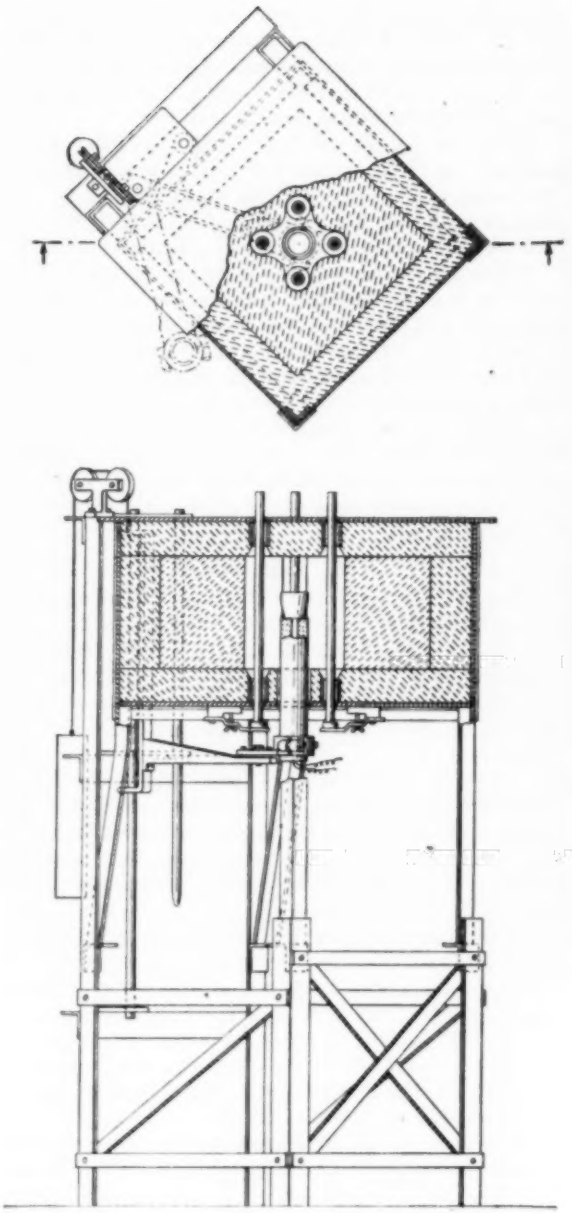

FIG. 1.—Diagram of electrically heated Globar furnace.

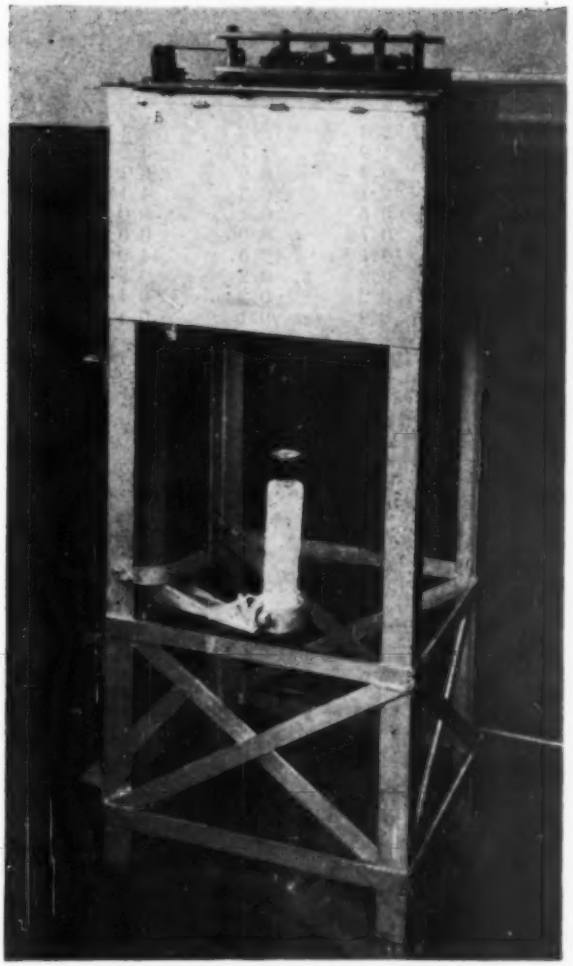

FIG. 2.—Photograph of furnace showing pier with crucible in lowered position.

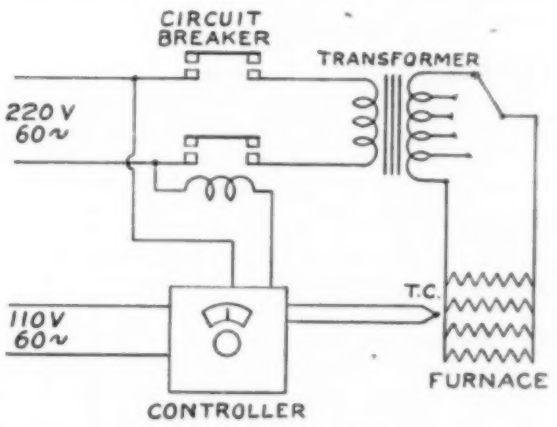

FIG. 3.—Wiring diagram.

A thermocouple, not shown in Fig. 1, extends through the side of the furnace at a point opposite the top of the crucible, and is connected to the on-off controller, which in turn is connected to the magnetic circuit breaker.

A portable potentiometer of high sensitivity and a standardized platinum-90 platinum 10% rhodium thermocouple are used for measuring the temperature of the crucible. The crucible is of chemically pure platinum with a capacity of 70 cc. and weighs approximately 65 gm.

Miscellaneous equipment includes carbon slabs and iron rings for the casting of samples, a small electric muffle furnace for annealing the samples, an inspection lamp, and an interval timer.

TABLE I

CHEMICAL ANALYSIS AND FINING-TIME DATA OF FOUR GLASSES

Oxide	Chemical analysis A	B	C	D
SiO_2	72.3	73.2	72.1	73.4
R_2O_3	1.0	1.7	3.0	2.1
CaO	6.9	8.6	6.1	5.0
MgO	2.1	0.2	4.1	3.4
BaO	0.5	0.4	0.0	0.0
B_2O_3	0.7	0.0	0.0	0.5
Na_2O	16.1	15.5	14.0	14.8
K_2O	0.2	0.2	0.6	0.5
SO_3	0.2	0.2	0.1	0.2
F_2	0.0	0.1	0.1	0.0
Total	100.0	100.1	100.1	99.9
°C.	Fining time (minutes)			
1400				175
1425	125	220	160	115
1445	85	150		
1450			100	80
1470	45			
1475		75	70	55

IV. Procedure

(1) Preparation of Batch

The batch materials are weighed in the desired proportions and in quantity sufficient to produce approximately 300 gm. of glass. The batch is mixed and ground in a pebble mill until all of it will pass a number 100 U. S. standard sieve.

(2) Preparation of Bubble-Free Bath

The furnace is heated to the temperature selected for making a test. The platinum crucible is charged with 65 gm. of batch and is placed on the pier in the furnace. From time to time, the crucible is lowered from the furnace, swung to the side, and examined through a blue glass filter. When the melt has become quiet and bubbles are no longer visible, the crucible is returned to the furnace for an additional fining period, during which the temperature is measured and final adjustments to the furnace are made.

(3) Measurement of Temperature

The standardized thermocouple is inserted through the hole in the pier and is held against the bottom of the crucible. The leads from the thermocouple are connected to the portable potentiometer and temperature readings are taken. The controller is adjusted, if necessary, to give the desired temperature. When it is certain that the desired test conditions have been established, the thermocouple is removed.

(4) Running the Test

Ten grams of batch are weighed and transferred to a small metal scoop. The crucible is lowered from the furnace, the charge of batch is added, and the crucible is quickly returned to the furnace. The interval timer is set for a test period judged to be less than required for the complete removal of bubbles. While the test is in progress, the annealing oven is heated to the estimated annealing temperature of the glass. At the end of the test period, the crucible is removed from the furnace by means of tongs, and, after the glass has cooled for a few seconds, a circular pat of glass about $1^1/_2$ in. in diameter and $^1/_4$ in. thick is cast. A preliminary examination for bubbles is made at this time.

When the sample has lost most of its color and is partially set, it is transferred to the annealing oven; the power is turned off and the sample is cooled. For final examination, the sample is illuminated from the side and is viewed against a dark background.

Following the casting of a sample, the glass in the crucible is replenished by addition of batch and the preparation of the bubble-free bath is repeated.

Additional tests are made using successively longer test periods until an end point is reached. As the end point is approached, the interval of the tests is adjusted to provide for an estimated precision of ±10% of the fining-time value. The shortest test interval required to produce a sample substantially free of bubbles is noted as the fining time of the glass at the corresponding temperature.

V. Comments

Temperatures employed in making determinations of fining times have ranged from 1400° to 1500°C. At temperatures less than 1400°C., the fining times are excessively long and the end points are uncertain. At temperatures greater than 1500°C., the useful life of heating elements and of platinum crucibles is greatly shortened.

Variations in the weight of batch used in the bath of molten glass and in the charge of batch added for the test are, within reasonable limits, without influence on the result. For this reason, it is possible to use constant weights of batch and to neglect corrections for the weights of glass produced.

Fining times ranging from 15 to 250 minutes have been determined with satisfactory precision. At 1475°C., the results obtained in tests of commercial glasses may be expected to lie between 25 and 75 minutes and at 1425°C., between 100 and 250 minutes.

The end point in a series of tests is usually marked by a sharp decline in the concentration, rather than by complete disappearance of bubbles. In nearly all tests, a few large bubbles persist at the edge of the melt and in contact with the side of the crucible. Two or three of these may be transferred in pouring the sample. Sometimes a string of very small bubbles is formed when the stream of glass is severed during the casting of the sample. With very little experience, it is possible to discount these defects in the final examination of the samples.

VI. Results

In Table I are listed the results from tests of fining times and the chemical analyses of four glasses selected as representative of compositions used in the manufacture of containers. The analyses list all of the constituents derived from the batch mixtures except arsenic, which was introduced as arsenious oxide at the rate of one part per thousand of sand in the batches for glasses A, B, and D, and at the rate of one and one-half parts for glass C.

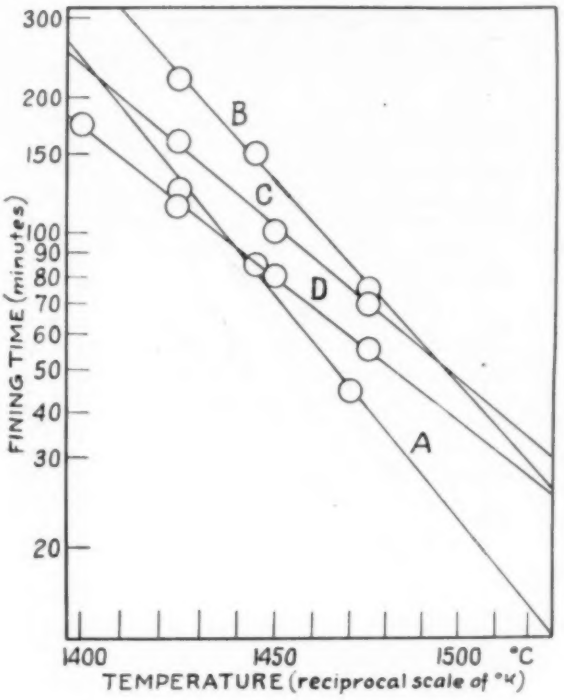

FIG. 4.—Fining time versus temperature.

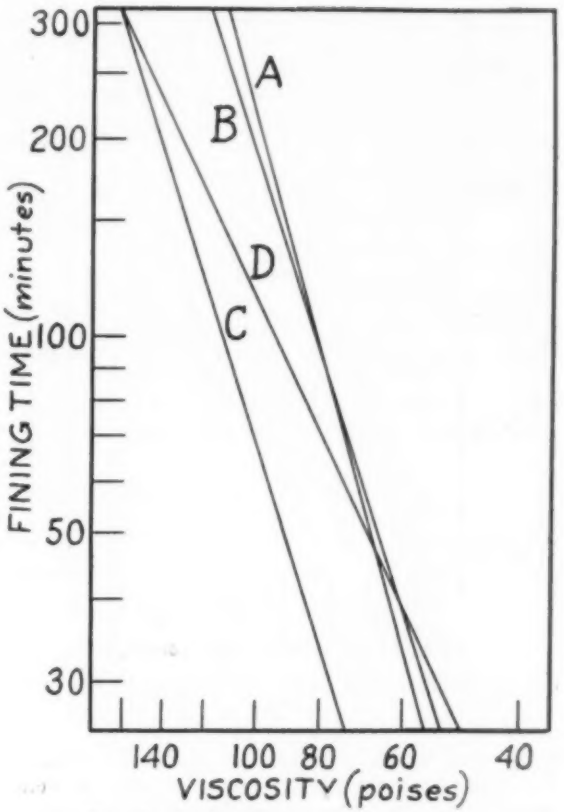

FIG. 5.—Fining time versus viscosity.

Inspection of the fining-time results reveals a wide range of values. Assuming, for the individual glasses, that fining time is related to temperatures by the equation of Arrhenius, the fining times have been plotted on a logarithmic scale against temperature on a scale of reciprocal degrees Kelvin. This is shown in Fig. 4. Straight lines give satisfactory representations of the data. For all practical purposes, it is permissible, in plotting the results to use a uniform scale of degrees centigrade for the range of temperatures normally employed.

Figure 4 demonstrates that the fining time must be determined for at least two temperatures and preferably three, if accurate comparisons between glasses are to be made. The random pattern of the lines makes this fact evident.

The commonly accepted opinion that fining is closely related to the viscosity of glass is not to be overlooked. In Fig. 5, the fining time is plotted against viscosity by the use of log-log coordinates. Viscosity data for the four glasses were obtained by extrapolation of results obtained at temperatures up to 1350°C. Even allowing for some inaccuracy in the extension of the viscosity curves, it is evident that fining times cannot be predicted from results of viscosity measurements and that there exists no more than a general correlation between the two properties. In fact, at constant viscosity, the ratio between extreme values for fining time is more than three to one while at constant temperature the ratio is only about two to one.

It has been suggested that surface tension is closely related to fining. Fining times of several simple soda-lime glasses were compared with published data[5] on surface tension, but again no simple relationship was evident.

The viscosity, surface tension, and density of the glass, the solubilities and the relative amounts of various gases in the batch and glass, and no doubt other factors affect the process of fining. In view of these many probable influences it is more desirable, from practical considerations, to define fining time in terms of a test method than it is to attempt correlation of the fining characteristics of glasses with one or two physical properties. It is essential, however, that the results obtained by the test be capable of practical interpretation.

VII. Interpretation of Results

Direct comparisons between commercial and laboratory results are difficult to make because in commercial practice the operating conditions are not easily defined nor is the degree of fining standardized. To provide a basis for direct comparisons, large-scale fining tests were conducted under carefully controlled conditions in

[5] (*a*) E. W. Washburn and G. R. Shelton, "Viscosities and Surface Tensions of Soda-Lime Glasses at High Temperatures: I, Viscosities at High Temperatures," *Univ. of Ill. Eng. Expt. Station Bull.*, No. 140, pp. 8–50 (April 14, 1924); (*b*) E. W. Washburn and E. E. Libman, "II, Surface Tensions of Glasses at High Temperatures," pp. 53–71; extended abstract, *Ceram. Abs.*, **3** [12] 336–44 (1924).

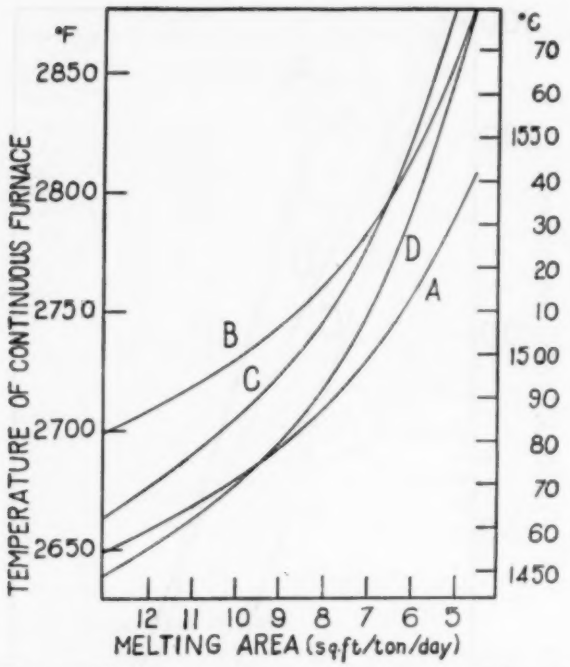

FIG. 6.—Melting area versus temperature of continuous furnace.

a day tank having a capacity of about five tons. It was found that the fining time in minutes, as determined in the laboratory, varied directly as the time required to produce fined glass in the day tank.

Further comparisons of fining times with data from records of continuous furnaces showed that the fining time could be correlated directly with the ratio of melting area to tons of glass produced. It was found also that the correlation could be made more nearly exact if a correction in temperature was made to allow for the difference in methods of measurement. The temperature of the continuous furnace was taken as the reading of an optical pyrometer sighted on the bridge wall just above the glass. The fining rate of a glass in a continuous furnace may be calculated from fining-time data by the use of equations (1) and (2).

$$A = 0.065F + 3.25 \quad (1)$$

$$t_c = 1.02t_l + 5 \quad (2)$$

A = melting area-tonnage ratio (sq. ft./ton/day).
F = fining time (minutes).
t_c = continuous furnace temp. (°C.).
t_l = laboratory furnace temp. (°C.).

Equation (1) appears to indicate a theoretical limit to a fining rate of 3.25 sq. ft. per ton per day. However, since even the most easily fined glass would require a temperature above the melting point of furnace refractories in order to approach this rate, no practical limitation is imposed by the equation.

Fining-time data, taken from Fig. 4 and equations (1) and (2), were used to establish the temperature-fining curves shown in Fig. 6. From these curves, temperatures and melting areas per ton for the four glasses of Table I can be read directly.

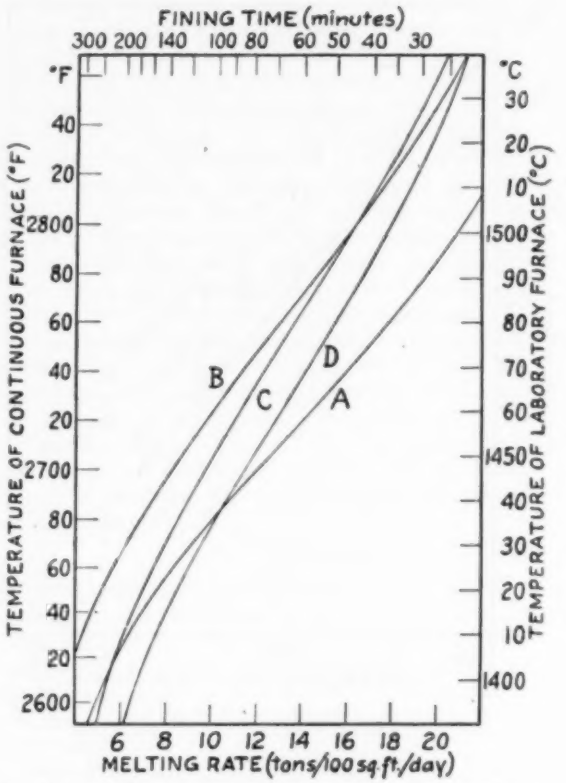

FIG. 7.—Melting rate versus temperature of continuous furnace.

Melting area, even though it is the most commonly employed designation of rate of production, is awkward to use in making comparisons. The reciprocal of melting area multiplied by 100 is the melting rate in tons per 100 sq. ft. per day and is much more convenient. In Fig. 7, the temperature-melting-rate curves are seen to be almost straight lines over the range of 10 to 18 tons per 100 sq. ft. The curves in Fig. 7 are for the same glasses as shown in Fig. 6. Several points are of interest.

Consider glasses A and D. At a melting rate of 10 tons, the two glasses require the same temperature. At 16 tons, which may be considered close to average practice, glass A requires a furnace temperature of 2740°F. while D requires 2770°F.

At very low rates, glasses A and C require about the same temperature, but as the melting rate is increased the difference in temperature becomes large. At 16 tons, glass C is fined at 2800°F. and glass A at a temperature 60° lower.

The temperature-melting-rate curves check closely with practice over the range of 12 to 20 tons per 100 sq. ft. A glass, similar in composition and in fining time to glass A, has been melted satisfactorily at a rate of 20 tons per 100 sq. ft. with a furnace temperature below 2780°F., but the glass was not completely fined. Glasses B and C, neither of which is now in commercial use, required furnace temperatures above 2800°F. for melting rates between 16 and 18 tons per 100 sq. ft.

Glass D, at 2650°F., has been melted and fined at the rate of 8.5 tons per 100 sq. ft. and at 2750° was not completely fined when melted at the rate of 16 tons per 100 sq. ft.

Differences in rates of fining, as great as those indicated in Fig. 7, may be of major importance in judging the operating efficiency of tank furnaces. Considering the extremes shown, which by no means include all the glasses measured, it is possible to account for a 10% range in fuel consumption or for a 30% range in melting rate.

VIII. Conclusions

The data given in this paper are taken from tests of colorless glasses, and the correlations established apply to furnaces supplying glass for the manufacture of containers. The method, however, is applicable to all types of glasses except carbon-sulfur ambers and other similar glasses made from batches which contain reducing agents. With slight modification, such as the substitution of ceramic crucibles for platinum, the method might also be extended to include reduced glasses.

The design of furnaces used for melting window and plate glasses is different from the design of container-glass furnaces, and the relative importance of bubbles is different in the two types of glass. It is unlikely that a single correlation formula would apply to all manufacturing conditions. A special formula should, therefore, be developed for each type of operation.

Other uses of the method for measuring fining time, in addition to that of determining the characteristics of existing glasses, include studies of the effectiveness of fining agents and investigations of the influence of composition on fining time. It is hoped that results obtained in systematic studies may lead to a better understanding of the process of fining, and eventually, to increased efficiency in the melting and fining of glasses.

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ANNEALING STRAINS IN GLASS AND THEIR DEGREE OF PERMANENCE*

Measurements of Optical Retardation over a Seven-Year Period on Glass Container Manufacturers' Standardized Disks

By L. G. Ghering and T. D. Green

Abstract

A statistical comparison has been made of 13 sets of measurements of optical path difference, taken over a seven-year period, on 24 samples of the standardized disks distributed in 1938 to the manufacturers of glass containers. These data show that the original optical retardation of about 22.8 millimicrons has not changed by more than ±1.3%, equivalent to ±0.30 millimicron or ±0.10° on the scale of the measuring instrument.

Five tests were made in 1938, five in 1945, and three between. The standard deviation of the combined errors for each of the 13 averages of 24 measurements is ±0.0338° and is composed of an accidental error of observation of ±0.005° and an error of adjustment of the instrument of ±0.0333°. The range of ±1.3%, or ±0.10°, beyond which the disks could not have changed over seven years, represents the 3-sigma probability limit of combined errors.

While this possible change of ±1.3% would be insignificant in relation to the utility of the standardized disks, it represents a possible experimental error too great to permit broad conclusions concerning the ultimate stability of birefringence in strained glass.

I. Introduction

In 1938 more than 1500 glass disks, showing identical strain patterns in the polariscope, were prepared by the Hartford-Empire Company laboratories for the Glass Container Association. These disks are used by the glass container manufacturing companies in sets of five (or seven) for the polariscopic examination of glass containers[1] and are commonly known as the GCA Standard Disks. The individual disks were cut from window glass and are 3.484 to 3.494 in. in diameter and approximately 0.092 in. thick. They have a deliberately introduced annealing stress† of approximately 510 p.s.i. resulting in approximately 22.8 millimicrons optical path difference for green light (mercury green line, 546.1 mμ) at points on a circle 0.250 in. from the edge of the disks.‡ The exact measurement for each individual disk was obtained by using a polarimeter which was essentially a duplicate of that described by Goranson and Adams.[2]

A description of the disks and the method of measuring them is in preparation[3] and a discussion of their use in grading containers for residual annealing stress has been given by Gooding.[4] The 22.8-millimicron retardation (optical path difference) corresponds to a 7.50° rotation of the analyzer of the polarimeter to give a null point, and it is this angular shift or displacement that is directly observed.§ The original measurements were observed and recorded by two observers, Green and Ghering, in the period January to July, 1938.

In order to investigate the permanence of the optical path difference of the standards in use by manufacturers, a sample of 36 disks was retained with the polarimeter at the Hartford-Empire Company laboratories. Subsequent measurements were to be made in hope of discerning any decrease in stress with time so that the validity of the standards after several years of aging would be known. Furthermore, it was hoped that the high precision of measurement attainable with this type of polarimeter would give data that would be useful in fundamental interpretations concerning the presence

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* Submitted to the Glass Division, The American Ceramic Society, for presentation at the Pittsburgh Local Section Autumn Meeting, October 12, 1945. Received June 22, 1945.

[1] Standard Method of Polariscopic Examination of Glass Containers, A.S.T.M. Designation C 148–43. A.S.T.M. Standards, 1944, Vol. II, pp. 362–64. American Society for Testing Materials, Philadelphia, Pa.

† Actually it is a known optical path difference, and it corresponds to a stress difference rather than stress. The stress computed from the optical retardation, which is 510 p.s.i., is the difference between the principal stresses at the point of measurement.

‡ In preparing the disks, a manufacturing tolerance of ±0.9 mμ was allowed so that disks measuring between 21.9 mμ and 23.7 mμ were accepted for use as standards.

[2] R. W. Goranson and L. H. Adams, "Method for Precise Measurement of Optical Path Difference, Especially in Stressed Glass," *Jour. Franklin Inst.*, **216** [4] 475–504 (1933); *Ceram. Abs.*, **13** [10] 254 (1934).

A description of the polarimeter used in these measurements has been recently prepared by Green (see footnote 3). Although Goranson and Adams claim a precision 100 times greater than that of devices commonly used, the angularly calibrated head and vernier used by the present authors reads to only 0.10° or 0.3 mμ per vernier division and is only about one fifth as sensitive as theirs.

[3] T. D. Green, "Preparation of Standardized Discs for the Strain Testing of Glassware." Submitted recently for presentation to the Society of Glass Technology (England).

[4] E. J. Gooding, "Use of Standard Discs in Strain Testing of Glassware," *Jour. Soc. Glass Tech.* (*Trans.*), **22**, 241–49 (1938); *Ceram. Abs.*, **18** [5] 126–27 (1939).

For a later report by Gooding on the same subject, see *Jour. Soc. Glass Tech.* (*Trans.*), **24**, 186–96 (1940).

§ Path difference = 3.034 mμ × scale reading in degrees for light of 546.1 mμ wave length. The sample of disks in this work averaged approximately 7.38° or 22.4 mμ path difference.

TABLE I

TABULATION OF TESTS AND SUMMARY OF DATA

Measurement sets	Date	Observer	No. of disks measured	Avg. reading of 1st 12 (degrees)		Avg. reading of all 36		Remarks
	1938							
1	January	Green	First 12	7.385				
2	March 15	"	" "	7.327				
3	May 20	"	All 36	7.423	7.434	7.420	7.430	Same adjustment of instrument (pair A)
4	May 31	"	" "	7.444		7.440		
5	January	Ghering	First 12	7.400				
6	July 7	"	All 36	7.412		7.416		
	1939							
7	April 28	Green	" "	7.308	7.314	7.288	7.292	Same adjustment of instrument (pair B)
8	" "	"	" "	7.319		7.296		
	1941							
9	November	"	First 12	7.365				
	1944							
10	August	"	" "	7.399				
	1945							
11	January 18	Ghering	" "	7.352				
12	January 22	"	All 36	7.390	7.390	7.397	7.400	Same adjustment of instrument (pair C)
13	" "	"	" "	7.390		7.402		
14	February 12	Green	" "	7.383	7.386	7.392	7.394	Same adjustment of instrument (pair D)
15	" "	"	" "	7.388		7.395		
16	February 28	"	First 12	7.358				
17	March 6	"	All 36	7.410	7.394	7.399	7.398	Same adjustment of instrument (pair E)
18	March 7	"	" "	7.379		7.397		

or absence of plastic flow in glass at room temperatures over long periods of time and, perhaps, even throw some light on the structure of glass.

II. Method of Measurement over a Seven-Year Period

Measurements on the aging test specimens were made in the same manner as those on the industry-distributed standards. Both maximum and minimum retardation were observed each time at points 0.250 in. from the edge, the maxima and minima being located independently each time by rotating the disk in the holder. Both values were recorded. The vernier on the calibrated head for measurement of angles was read to the nearest 0.05°, for example, 7.45°, 7.50°, or 7.55°. Because of the small angle being measured, the precision of reading the angular scale is no greater than approximately 0.7%.*

Before each series of measurements, the holder was reset and checked so that the center of the beam of light would pass through the disk 0.250 in. from the edge. Furthermore, in most instances some optical parts of the instrument were removed, cleaned, and then readjusted to give an exact zero reading on the scale (with no specimen in the holder). The readjustment of the holder, however, has probably been the largest component of the error of instrument adjustment. The magnitude of the instrument error will be discussed in connection with the interpretation of the data.

The original ring holder was made of plastic for low thermal conductivity. In retrospect, this was unfortunate, because it did not remain true in dimensions during the seven-year period, and it was necessary to use a new "wheel" holder of improved design in some of the later measurements. The ring holder, moreover, originally differed slightly from the correct dimensions so that a small known correction has been applied to the data. This correction, applied to the averages and amounting to 0.0250°, appears in the data whenever the original plastic ring was used.

Inasmuch as there was an interval of days, weeks, or sometimes months between observations by the two observers, the instrument was in all instances readjusted between observers, and it is likely that any small difference between observers is in reality a difference in setting or adjustment of the instrument.

For measuring the industry-distributed standards, it was found necessary to have the disk under strict temperature equilibrium during measurement. An error of +0.25° could occur if the instrument and the storage tray for the samples differed in temperature by a few degrees. Accordingly, for each measurement on the aging-test specimens, care was taken to insure temperature equilibrium, either by allowing the disk to remain in the holder 10 to 30 minutes before an observation or by maintaining equality between the temperature of the stage and that of the storage tray and making occasional checks for temperature effect. In any case, care was taken to maintain a constant temperature in the room and to avoid drafts.

* There is evidence that the total fluctuation of other components of the polarimeter observations, namely, the matching of the two halves of the field, the adjustment of the vernier in making the null point balance, and the reading of the vernier, is somewhat less than 0.05°, possibly 0.02°. When these operations are made five times in a row for the zero point (no specimen) or for a particular specimen, it is rare for the value 7.50°, for example, to change to 7.45° or 7.55° on any of the five readings. The value, 0.02°, corresponds to approximately 0.06 mμ retardation.

TABLE II

POLARIMETER READINGS (IN DEGREES) ON 12 SAMPLE DISKS (Nos. A112 TO A699)

Measurement No.		1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18
				1938				1939		1941	1944					1945			
Specimen	Date	Jan.*	Mar.*	May*	May*	Jan.†	July†	Apr.*	Apr.*	Nov.*	Aug.*	Jan.†	Jan.†	Jan.†	Feb.*	Feb.*	Feb.*	Mar.*	Mar.*
A112	Max.	7.45	7.35	7.40	7.40	7.50	7.35	7.30	7.30	7.40	7.40	7.30	7.40	7.40	7.40	7.50	7.40	7.45	7.40
	Min.	7.30	7.20	7.30	7.30	7.40	7.25	7.20	7.20	7.25	7.30	7.20	7.30	7.25	7.30	7.30	7.25	7.30	7.30
A165	Max.	7.30	7.30	7.40	7.35	7.30	7.45	7.25	7.30	7.20	7.40	7.30	7.35	7.35	7.40	7.35	7.30	7.35	7.35
	Min.	7.20	7.15	7.20	7.30	7.20	7.25	7.10	7.10	7.15	7.20	7.10	7.20	7.25	7.20	7.20	7.15	7.20	7.15
A228	Max.	7.25	7.10	7.20	7.30	7.20	7.25	7.15	7.10	7.15	7.25	7.15	7.25	7.25	7.30	7.20	7.15	7.20	7.20
	Min.	7.20	7.10	7.10	7.20	7.15	7.15	7.05	7.05	7.10	7.15	7.10	7.20	7.15	7.10	7.15	7.10	7.20	7.15
A278	Max.	7.40	7.35	7.40	7.40	7.35	7.35	7.35	7.35	7.35	7.30	7.35	7.35	7.40	7.35	7.35	7.30	7.40	7.30
	Min.	7.20	7.15	7.30	7.30	7.25	7.25	7.15	7.25	7.30	7.20	7.30	7.25	7.30	7.25	7.30	7.20	7.30	7.25
A327	Max.	7.30	7.30	7.40	7.40	7.40	7.35	7.30	7.30	7.35	7.40	7.35	7.35	7.40	7.40	7.40	7.30	7.45	7.30
	Min.	7.20	7.15	7.20	7.30	7.30	7.20	7.20	7.15	7.20	7.20	7.25	7.25	7.30	7.30	7.20	7.20	7.25	7.20
A377	Max.	7.60	7.50	7.55	7.70	7.60	7.55	7.50	7.50	7.70	7.60	7.55	7.65	7.60	7.60	7.60	7.55	7.60	7.55
	Min.	7.30	7.40	7.45	7.45	7.35	7.40	7.35	7.35	7.40	7.40	7.40	7.45	7.45	7.40	7.40	7.40	7.50	7.45
A433	Max.	7.65	7.60	7.75	7.70	7.65	7.70	7.60	7.60	7.70	7.67	7.65	7.70	7.70	7.70	7.70	7.70	7.70	7.65
	Min.	7.50	7.50	7.60	7.60	7.50	7.55	7.40	7.40	7.50	7.55	7.50	7.55	7.55	7.50	7.55	7.45	7.50	7.50
A483	Max.	7.60	7.50	7.60	7.70	7.55	7.60	7.45	7.50	7.60	7.55	7.55	7.60	7.55	7.60	7.60	7.55	7.60	7.60
	Min.	7.50	7.40	7.50	7.50	7.40	7.50	7.40	7.45	7.50	7.50	7.50	7.50	7.45	7.45	7.50	7.50	7.50	7.50
A550	Max.	7.50	7.45	7.60	7.60	7.60	7.55	7.40	7.45	7.50	7.60	7.55	7.55	7.55	7.55	7.60	7.55	7.50	7.55
	Min.	7.15	7.20	7.35	7.30	7.40	7.35	7.20	7.20	7.25	7.20	7.30	7.30	7.35	7.30	7.25	7.30	7.30	7.30
A601	Max.	7.50	7.40	7.50	7.55	7.45	7.45	7.40	7.40	7.45	7.50	7.50	7.55	7.50	7.50	7.50	7.40	7.45	7.50
	Min.	7.35	7.20	7.35	7.40	7.35	7.35	7.20	7.20	7.30	7.40	7.30	7.35	7.30	7.30	7.30	7.30	7.30	7.30
A649	Max.	7.20	7.10	7.30	7.20	7.20	7.25	7.10	7.10	7.10	7.20	7.20	7.20	7.20	7.20	7.20	7.10	7.15	7.15
	Min.	7.10	7.00	7.10	7.10	7.10	7.15	7.00	7.00	7.10	7.10	7.05	7.10	7.10	7.10	7.15	7.00	7.10	7.10
A699	Max.	7.60	7.50	7.60	7.60	7.50	7.60	7.45	7.50	7.40	7.50	7.60	7.55	7.60	7.60	7.60	7.50	7.55	7.60
	Min.	7.30	7.35	7.40	7.40	7.30	7.45	7.30	7.30	7.20	7.40	7.40	7.40	7.40	7.40	7.40	7.35	7.40	7.35
Avg. Correction		7.360 +0.025	7.302 +0.025	7.398 +0.025	7.419 +0.025	7.375 +0.025	7.387 +0.025	7.283 +0.025	7.294 +0.025	7.340 +0.025	7.374 +0.025	7.352	7.390	7.390	7.383	7.388	7.333 +0.025	7.385 +0.025	7.354 +0.025
				7.423	7.444			7.308	7.319				7.390	7.390	7.383	7.388		7.410	7.379
Corrected avg.		7.385	7.327	7.434		7.400	7.412	7.314		7.365	7.399	7.352	7.390		7.386		7.358	7.394	

* Observer Green.
† Observer Ghering.

TABLE III

FIVE PAIRS OF DUPLICATE OBSERVATIONS ON ALL 36 SAMPLE DISKS (IN DEGREES)

		Pair A, Green			Pair B, Green			Pair C, Ghering			Pair D, Green			Pair E, Green		
Specimen		3	4	Diff.	7	8	Diff.	12	13	Diff.	14	15	Diff.	17	18	Diff.
A112	Max.	7.40	7.40		7.30	7.30		7.40	7.40		7.40	7.50	0.10	7.45	7.40	0.05
	Min.	7.30	7.30		7.20	7.20		7.30	7.25	0.05	7.30	7.30		7.30	7.30	
A165	Max.	7.40	7.35	0.05	7.25	7.30	0.05	7.35	7.35		7.40	7.35	.05	7.35	7.35	
	Min.	7.20	7.30	.10	7.10	7.10		7.20	7.25	.05	7.20	7.20		7.20	7.15	.05
A228	Max.	7.20	7.30	.10	7.15	7.10	.05	7.25	7.25		7.30	7.20	.10	7.20	7.20	
	Min.	7.10	7.20	.10	7.05	7.05		7.20	7.15	.05	7.10	7.15	.05	7.20	7.15	.05
A278	Max.	7.40	7.40		7.35	7.35		7.35	7.40	05	7.35	7.35		7.40	7.30	.10
	Min.	7.30	7.30		7.15	7.25	.10	7.25	7.30	.05	7.25	7.30	.05	7.30	7.25	.05
A327	Max.	7.40	7.40		7.30	7.30		7.35	7.40	.05	7.40	7.40		7.45	7.30	.15
	Min.	7.20	7.30	.10	7.20	7.15	.05	7.25	7.30	.05	7.30	7.20	.10	7.25	7.20	.05
A377	Max.	7.55	7.70	.15	7.50	7.50		7.65	7.60	.05	7.60	7.60		7.60	7.55	.05
	Min.	7.45	7.45		7.35	7.35		7.45	7.45		7.40	7.40		7.50	7.45	.05
A433	Max.	7.75	7.70	.05	7.60	7.60		7.70	7.70		7.70	7.70		7.70	7.65	.05
	Min.	7.60	7.60		7.40	7.40		7.55	7.55		7.50	7.55	.05	7.50	7.50	
A483	Max.	7.60	7.70	.10	7.45	7.50	.05	7.60	7.55	.05	7.60	7.60		7.60	7.60	
	Min.	7.50	7.50		7.40	7.45	.05	7.50	7.45	.05	7.45	7.50	.05	7.50	7.50	
A550	Max.	7.60	7.60		7.40	7.45	.05	7.55	7.55		7.55	7.60	.05	7.50	7.55	.05
	Min.	7.35	7.30	.05	7.20	7.20		7.30	7.35	.05	7.30	7.25	.05	7.30	7.30	

TABLE III (*concluded*)

Specimen		Pair A, Green			Pair B, Green			Pair C, Ghering			Pair D, Green			Pair E, Green		
		3	4	Diff.	7	8	Diff.	12	13	Diff.	14	15	Diff.	17	18	Diff.
A601	Max.	7.50	7.55	0.05	7.40	7.40		7.55	7.50	0.05	7.50	7.50		7.45	7.30	0.05
	Min.	7.35	7.40	.05	7.20	7.20		7.35	7.30	05	7.30	7.30		7.30	7.30	
A649	Max.	7.30	7.20	.10	7.10	7.10		7.20	7.20		7.20	7.20		7.15	7.15	
	Min.	7.10	7.10		7.00	7.00		7.10	7.10		7.10	7.15	0.05	7.10	7.10	
A699	Max.	7.60	7.60		7.45	7.50	0.05	7.55	7.60	.05	7.60	7.60		7.55	7.60	.05
	Min.	7.40	7.40		7.30	7.30		7.40	7.40		7.40	7.40		7.40	7.35	.05
A734	Max.	7.55	7.50	.05	7.45	7.45		7.55	7.60	.05	7.60	7.60		7.50	7.50	
	Min.	7.35	7.30	.05	7.15	7.20	.05	7.30	7.40	.10	7.30	7.30		7.30	7.30	
A785	Max.	7.50	7.50		7.35	7.35		7.50	7.50		7.50	7.50		7.50	7.50	
	Min.	7.35	7.40	.05	7.15	7.20	.05	7.30	7.35	.05	7.30	7.30		7.30	7.30	
A835	Max.	7.50	7.50		7.40	7.45	.05	7.55	7.55		7.50	7.60	.10	7.50	7.50	
	Min.	7.40	7.50	.10	7.30	7.35	.05	7.45	7.45		7.40	7.40		7.40	7.40	
A885	Max.	7.70	7.65	.05	7.50	7.45	.05	7.75	7.65	.10	7.70	7.75	.05	7.70	7.60	.10
	Min.	7.50	7.50		7.30	7.30		7.50	7.50		7.45	7.45		7.50	7.45	.05
A935	Max.	7.60	7.55	.05	7.40	7.45	.05	7.55	7.55		7.60	7.55	.05	7.50	7.55	.05
	Min.	7.40	7.30	.10	7.30	7.25	.05	7.30	7.35	.05	7.30	7.30		7.30	7.30	
A984	Max.	7.35	7.40	.05	7.30	7.25	.05	7.35	7.40	.05	7.40	7.40		7.35	7.35	
	Min.	7.15	7.20	.05	7.05	7.05		7.15	7.20	.05	7.20	7.15	.05	7.20	7.20	
A1035	Max.	7.50	7.50		7.35	7.30	.05	7.45	7.50	.05	7.50	7.45	.05	7.40	7.35	.05
	Min.	7.30	7.35	.05	7.15	7.20	.05	7.25	7.30	.05	7.20	7.30	.10	7.30	7.30	
A1085	Max.	7.40	7.50	.10	7.30	7.30		7.50	7.45	.05	7.50	7.50		7.50	7.50	
	Min.	7.15	7.25	.10	7.00	7.00		7.20	7.20		7.20	7.15	.05	7.25	7.20	.05
A1135	Max.	7.35	7.40	.05	7.30	7.30		7.35	7.40	.05	7.40	7.40		7.35	7.40	.05
	Min.	7.10	7.10		7.00	7.00		7.15	7.15		7.10	7.10		7.10	7.15	.05
A1185	Max.	7.50	7.50		7.30	7.35	.05	7.45	7.50	.05	7.50	7.50		7.40	7.45	.05
	Min.	7.40	7.40		7.20	7.20		7.35	7.40	.05	7.40	7.35	.05	7.40	7.30	.10
A1235	Max.	7.50	7.60	.10	7.40	7.35	.05	7.55	7.55		7.55	7.60	.05	7.45	7.55	.10
	Min.	7.30	7.45	.15	7.20	7.20		7.35	7.35		7.30	7.35	.05	7.30	7.30	
A1285	Max.	7.30	7.30		7.15	7.15		7.30	7.30		7.30	7.30		7.30	7.25	.05
	Min.	7.15	7.20	.05	7.00	7.00		7.20	7.15	.05	7.15	7.15		7.15	7.20	.05
A1335	Max.	7.50	7.50		7.30	7.30		7.45	7.45		7.50	7.50		7.40	7.40	
	Min.	7.40	7.40		7.20	7.20		7.35	7.40	.05	7.35	7.40	.05	7.35	7.40	.05
A1388	Max.	7.40	7.35	.05	7.20	7.20		7.35	7.40	.05	7.30	7.35	.05	7.30	7.30	
	Min.	7.30	7.30		7.15	7.10	.05	7.25	7.30	.05	7.30	7.30		7.20	7.25	.05
A1402	Max.	7.60	7.60		7.45	7.45		7.65	7.65		7.60	7.65	.05	7.60	7.55	.05
	Min.	7.45	7.50	.05	7.30	7.35	.05	7.50	7.45	.05	7.50	7.45	.05	7.50	7.45	.05
A1450	Max.	7.40	7.40		7.25	7.20	.05	7.45	7.40	.05	7.45	7.40	.05	7.35	7.35	
	Min.	7.20	7.25	.05	7.10	7.10		7.30	7.25	.05	7.20	7.20		7.20	7.20	
A1500	Max.	7.50	7.45	.05	7.30	7.35	.05	7.50	7.50		7.50	7.50		7.45	7.40	.05
	Min.	7.35	7.35		7.20	7.20		7.35	7.30	.05	7.35	7.30	.05	7.30	7.30	
A1550	Max.	7.50	7.50		7.35	7.35		7.45	7.50	.05	7.40	7.40		7.40	7.50	.10
	Min.	7.30	7.30		7.10	7.15	.05	7.25	7.30	.05	7.30	7.30		7.25	7.25	
A1600	Max.	7.50	7.60	.10	7.45	7.50	.05	7.55	7.65	.10	7.60	7.60		7.55	7.60	.05
	Min.	7.40	7.40		7.20	7.25	.05	7.40	7.40		7.40	7.40		7.40	7.45	.05
A1650	Max.	7.60	7.50	.10	7.35	7.40	.05	7.50	7.55	.05	7.50	7.50		7.45	7.50	.05
	Min.	7.40	7.40		7.20	7.20		7.40	7.35	.05	7.35	7.35		7.40	7.35	.05
A1679	Max.	7.35	7.40	.05	7.20	7.20		7.30	7.30		7.30	7.30		7.20	7.30	.10
	Min.	7.20	7.20		7.10	7.10		7.20	7.20		7.15	7.20	.05	7.15	7.20	.05
A1730	Max.	7.50	7.60	.10	7.50	7.50		7.60	7.60		7.60	7.65	.05	7.55	7.60	.05
	Min.	7.30	7.30		7.15	7.20	.05	7.30	7.25	.05	7.30	7.30		7.30	7.30	
A1765	Max.	7.50	7.50		7.35	7.30	.05	7.50	7.50		7.50	7.45	.05	7.30	7.40	.10
	Min.	7.25	7.25		7.10	7.10		7.30	7.20	.10	7.25	7.20	.05	7.20	7.20	
A1800	Max.	7.60	7.60		7.45	7.40	.05	7.55	7.60	.05	7.60	7.60		7.50	7.55	.05
	Min.	7.45	7.45		7.20	7.20		7.45	7.40	.05	7.40	7.40		7.40	7.35	.05
Total				2.75			1.60			2.40			1.80			1.65
Avg.		7.395	7.415	0.0382	7.263	7.271	0.0222	7.372	7.377	0.0333	7.392	7.395	0.0250	7.374	7.372	0.0229
Correction		+0.025	+0.025		+0.025	+0.025		+0.025	+0.025					+0.025	+0.025	
Corrected avg.		7.420	7.440		7.288	7.296		7.397	7.402		7.392	7.395		7.399	7.397	

No definite time schedule for measurements on the aging-test specimens was fixed in advance. The recent measurements in 1945, however, were deliberately made in the same months of the year (January through March) as those of 1938 in order to avoid seasonal temperature differences. The 1938 and 1945 measurements moreover are seven years apart, within a month or two.

At each time of measurement, at least 12 disks were measured in order to increase the precision of observation, or, loosely speaking, to average out the accidental errors of observation. Both maximum and minimum of each of the 12 disks were observed, and the resulting 24 readings were arranged in a single column as if they were 24 single readings on 24 specimens; thus the averages reported are for samples of 12 disks and samples of 24 readings. The precision (standard deviation) of the average with respect to the accidental error of observation is the precision of a single observation divided by the square root of the number of observations, that is,

$$\sigma_{avg.} = \frac{\sigma'}{\sqrt{n}}$$

Since it is necessary to quadruple the number of observations in order to double the precision of the average and since a sample of 24 reduces the error of the average to approximately one fifth that of the single observation, it was not considered worth while to measure more than 12 disks each time, although in some instances all 36 were measured.

III. Data

A tabulation of the various sets of measurements and a summary of the data are given in Table I. In the 18 sets, there are five pairs of duplicate sets, namely, Nos. 3 and 4, 7 and 8, 12 and 13, 14 and 15, and 17 and 18, where both members of a particular pair were observed by the same observer, either on the same day or within one or two days, with no intervening readjustment of the instrument. The duplication on the same setting of the instrument is especially important, as will be shown in section IV(2), in that it makes it possible to distinguish between error of the instrument adjustment and error of observation. In addition to the five pairs of duplicate sets, there are eight other sets of measurements making a total of 13 independent measurements, independent in the sense that they correspond to 13 independent adjustments of the instrument. Of these adjustments, five were made in the year 1938, one in 1939, one in 1941, one in 1944, and five in 1945; furthermore, nine were by Green and four were by Ghering.

The 18 sets of measurements are listed in Table II, giving the maximum and minimum of each of the first 12 specimens, a total of 24 readings in each set of measurements. The 13 averages, with corrections for the dimensional error of the ring holder of $+0.025°$ where indicated, are listed at the bottom of Table II and are plotted in Fig. 1. These 13 averages represent the 13 independent adjustments.

The five pairs of duplicate sets on all 36 specimens are given in Table III. The second column of a particular pair consists of readings taken at the same instrument adjustment as the first column of the pair. The different pairs are at different adjustments. Pair C was observed by Ghering; the others by Green.

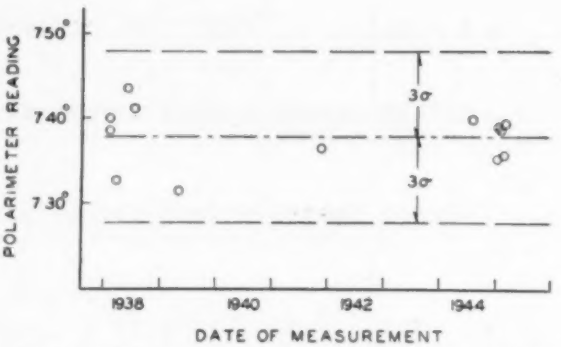

FIG. 1.—Averages of 13 sets of readings on 12 disks; central line is grand average; 3σ limits include errors of adjustment and errors of observation.

IV. Analysis and Discussion of Data

(1) Preliminary Analysis of Effects of Observers and Time

Prior to the completion of the measurement of series Nos. 16, 17, and 18, a preliminary analysis of variance[5] was made in order to separate the effects of (*a*) observer, (*b*) time, (*c*) "duplicate" tests by the same observer at different adjustment within the relatively short time of about six months, (*d*) real differences between specimens, and (*e*) accidental error of observation. Eight sets of measurements were used, namely, Nos. 1, 3, 5, and 6 (two for each observer in 1938) and 11, 12, 14, and 15 (two for each observer in 1945). This analysis showed that there was no statistically significant difference with time but that there was a significant difference between observers, between tests by the same observer taken within a six months' period, and between the specimens. Of course, the latter is seen from an inspection of the data and is immaterial as long as the same set of specimens is used. This preliminary work merely confirmed what was already suspected, namely, that two tests by the same observer within a relatively short time are not reproducible within the accidental error of observation and therefore the real variable is something other than time, probably the adjustment of the instrument.

(2) Accidental Error of Observation

In order to evaluate the error of instrument adjustment, it is necessary first to evaluate the accidental error of observation. For this purpose, the accidental error must be distinguished from the systematic error arising in the adjustment of the instrument. The accidental error arose from three operations, namely, (*a*) locating the position of maximum or minimum reading on the disk, (*b*) achieving the null setting of the analyzer

[5] (*a*) P. R. Rider, Modern Statistical Methods, pp. 137–50. John Wiley & Sons, New York, 1939.
(*b*) George Snedecor, Statistical Methods. Iowa State College Press, Ames, Iowa.

and (*c*) reading the scale. This accidental error appears between individual readings, and between the averages, of paired sets of readings in Tables II and III. It also exists among the readings in a particular column of data but is obscured by real differences among the specimens.

The systematic error, which arose in adjustment of the instrument, particularly of the disk holder, appears between the 13 averages (which include the five grand averages of the paired readings) in Table II.

The accidental errors were random or chance fluctuations, and as such tended to disappear from the averages as more and more readings were taken. The systematic errors remained fixed and equally applicable to all individual readings and to averages as long as the instrument adjustment remained unchanged.

In the ideal physical experiment, errors in the adjustment of the instrument other than those of resetting the holder would have been greatly reduced by calibration through reference to some permanent standard of retardation. Such a standard could be a quartz plate ground to a thickness providing optical retardation approximately equal to that of the disks. In the present work unfortunately, no such reference standard was available.*

The magnitude of the accidental error of observation can be estimated from the five pairs of duplicate sets of measurements in Table III. In all pairs except A and D, the first column was completed before the second was started and, of course, the results of the first were not at hand nor were they remembered as the second was being taken. The statistical method of computing the accidental error from such blind "duplicate" measurements on several specimens where the specimens differ among themselves is given by Simon.[6] It consists essentially in computing the standard deviation of the numerical values of the 72 differences and dividing by the square root of two, since two observations make up each difference. These differences and their averages are included in Table III. The five estimates turn out to be 0.0312°, 0.0187°, 0.0206°, 0.0223°, and 0.0284°, and the average value, 0.024°, is taken as the best estimate of the standard deviation of the accidental error of observation.† Since this applies to an individual observation, the standard deviation for averages of 24 is approximately ± 0.005 $(0.024/\sqrt{24})$ and the 3-sigma limits between which 99.7% of all averages of 24 readings would be found are ±0.015°. For averages of 72, the 3-sigma limits are approximately ±0.009° $(3 \times 0.024/\sqrt{72})$; therefore averages of 24 will lie within ±0.015° and averages of 72 within ±0.009°.

Referring to Table I, the averages of 36 specimens (72 readings) as shown vary from 7.288° to 7.440°, which is well outside a band of ±0.009 and indicates clearly that something other than accidental error of observation is involved. The averages of 12 specimens (24 readings) also vary from 7.314° to 7.434°, which is outside ±0.015°. There is reason to believe, therefore, that the adjustment of the instrument is an important variable, which will be discussed in connection with the question of aging.

(3) Aging

In Fig. 1, the averages representing the 13 independent adjustments of the instrument are plotted with time. These are averages of 24 readings taken from the last line of Table II. For each of the five pairs of duplicates, the combined average is used. Inspection of the figure shows that there is no systematic trend with time. The average of the five 1938 measurements (7.392°), moreover, is about the same as the average of the five 1945 measurements (7.372°), and the total spread of 7.314° to 7.434° over the seven years is not much greater than the spread of 7.327° to 7.434° for the year 1938 alone. The standard deviation for the five 1938 averages is approximately 0.04° and all values in subsequent years are within ±2 standard deviations. It is evident, then, that changes in the instrument adjustments within a particular year can entirely account for scatter of the points in Fig. 1, and the derivation of any limits, beyond which it may be said that the stress in the disks definitely has not changed, must be based on probability limits of the variability of the instrument adjustment.‡ In view of the fact that the preliminary analysis of variance mentioned in section IV (1) showed no statistically significant change with time and also because the 1938 and 1945 averages are practically the same (7.392° and 7.372°), the actual change may be assumed to be zero and the grand average may be used as the "trend" line in Fig. 1. Probability limits can then be applied to the horizontal trend line.

(4) Variability of Instrument Adjustment

On the assumption, based on the above evidence, that time is not a significant variable, the standard deviation and 3-sigma probability limits of the instrument adjustments may be computed. For this purpose, the 13 independent adjustments over the seven years are available. Using the 13 averages of 24 readings in Table II and Fig. 1, the squared standard deviation turns out to be 0.001139° and the standard deviation 0.0338°. This includes both the systematic (instrument adjustment) error and the accidental error. In order to isolate the systematic error from the accidental error, it is necessary to subtract from the squared standard deviation of 0.001139 for the total error the squared

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* The error of locating the disk holder was probably reduced in the 1945 measurements by the use of the wheel holder of improved design.

[6] L. E. Simon, Engineers' Manual of Statistical Methods, p. 155. John Wiley & Sons, New York, 1941.

† This value of 0.024° is an unbiased estimate of the standard deviation and $(0.024)^2$ is an unbiased estimate of the squared standard deviation. The total sum of the squares of the deviations of the five sets of 72 differences is 0.43080. Dividing by 360 gives 0.001197, or by 359 ($n - 1$ for an unbiased estimate) gives 0.001200. Extracting the square root and dividing by the square root of two gives 0.0244 in either case. The value is rounded off to 0.024.

‡ The standard deviation for the five 1945 measurements is considerably less than 0.04°, which probably represents an improvement in technique although it might be chance

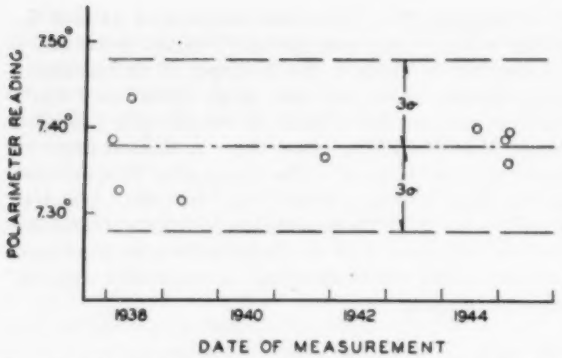

FIG. 2.—Average of 9 sets of readings by Green; other details same as Fig. 1.

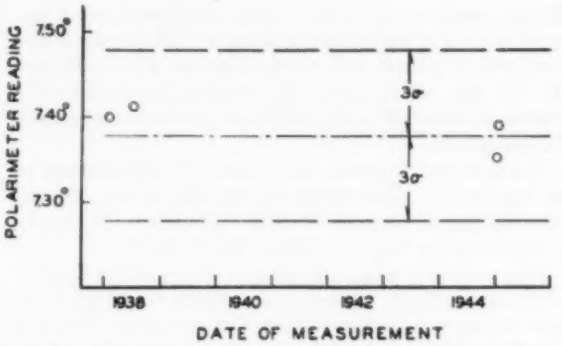

FIG. 3.—Averages of 4 sets of readings by Ghering; other details same as Fig. 1.

standard deviation for the accidental error.* The latter value is 0.000024, that is $(0.024)^2/24$. The remainder then is 0.0011115, which is the squared standard deviation of the systematic error. The square root is 0.0333°, which is the standard deviation of the systematic (instrument adjustment) error. These statistical results are summarized in Table IV, where the 3-sigma limits also are tabulated.

In comparing the combined errors with the instrument error alone in lines (A) and (C) of Table IV, the standard deviations differ very little, namely, 0.0338° compared with 0.0333°. This small difference arises from the fact that only squared standard deviations (variances), and not the standard deviations, are additive.

The 3-sigma limits of ±0.1014° in line (A) of Table IV are plotted in Fig. 1. As discussed in section IV (3), the central line representing the grand average of 7.378° is drawn in horizontally on the presumption that there is no change in the disks with time.† The 3-sigma band defines the limits beyond which the disks almost certainly have not changed and within which the sum of all errors may be expected to fluctuate.

(5) Comparison of Observers

In Fig. 2, only Green's averages of the first 12 specimens are plotted; and in Fig. 3, only Ghering's. The central lines and 3-sigma limits are the same as those used in Fig. 1. The difference between the average of Green's nine measurements (7.374°) and that of Ghering's 4 measurements (7.389°) is 0.015° which, from inspection of Figs. 2 and 3, appears to be a statistical fluctuation depending on the adjustments of the instrument rather than a real difference between observers. No better comparison is possible, inasmuch as no paired measurements were made by the two observers without intervening readjustments.

TABLE IV

SUMMARY OF STATISTICS

	Kind of error	Computed from	Squared standard deviation	Sigma standard deviation	3-sigma limits
(A)	Combined (accidental and systematic of avg. of 24)	13 sets (Table II)	0.001139	0.0338	0.1014
(B)	Accidental (observation of avg. of 24)	D (Table IV)	.000024	.005	.015
(C)	Systematic (instrument adjustments of avg. of 24)	A–B (Table IV)	.001115	.0333	.0999
(D)	Accidental (individual readings)	Pairs A–E (Table III)	.000576	.024	.072
(E)	Accidental (avg. of 72)	D (Table IV)	.000008	.003	.009

(6) Stability of Standards

As discussed previously, the experimental errors of measurement have a 3-sigma probability limit of approximately ±1.3%, equivalent to ±0.30 millimicron retardation. The definite statement can be made that the change due to aging over seven years has not been greater than the full range of 2.6%; it has probably been less, but it is not possible to estimate how much less from the data alone. Using these figures of ±1.3% or ±0.30 millimicron as the maximum possible change, it is clear that seven years' aging has not affected the validity of the standards, since changes of several times this amount are barely visible in the ordinary polariscope in the use of the standards. This maximum amount of change of ±2.6% is too great, however, for making conclusions concerning the stability of the an-

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* See Simon, p. 158 (footnote 6 of this paper). Note the two terms in the formula for σ^2_s at the top of this page.

$$\sigma^2_s = \sum \frac{(\bar{x} - \bar{\bar{x}})^2}{k-1} - \frac{\sigma^2_a}{n}$$

σ_s = standard deviation of systematic error.
σ_a = standard deviation of accidental error.

In the present application, the first term is the total sum of the squares of deviations of the 13 averages, in relation to the grand average, divided by 12, i.e., by $(k-1)$, where $k = 13$ and is equal to 0.001139, the total squared standard deviation. The second term is the squared standard deviation of the accidental error of observation for individuals divided by the number of observations. The accidental error in the present case is 0.024° for individuals (see section IV (2)) and the number of observations (n) is 24.

† This is the grand average of the 13 averages in the bottom line of Table II. If all 18 averages are used, giving, in effect, double weight to the five adjustments where duplicate measurements were made, the grand average would become 7.379° instead of 7.378°.

nealing stress. A possible change of 2.6% indicates a (remotely) possible deterioration to zero after about 270 years.* The order of accuracy of this work is therefore too low to permit deductions concerning the absence or presence of plastic flow, or other phenomena related to ultimate physical properties, in glass at room temperature.

V. Summary

(1) A sample of 36 disks from the industry-distributed standards has been measured in five different years by two different observers over a seven-year period in order to determine whether or not there has been any change with time in the deliberately introduced optical path difference due to a residual annealing stress of approximately 510 p.s.i.

(2) Each set of measurements at a given time included at least 24 readings with a standard deviation of the accidental error of observation of ±0.025° for an individual reading and ±0.005° for an average of 24.

(3) The average of several sets of measurements within a six months' period in a particular year was found to have a standard deviation much greater than ±0.005° and this larger error was found to be due to the adjustment of the polarimeter, in particular the adjustment of the holder which was readjusted at each time of measurement. The average value, however, obtained with five adjustments in 1938 was 7.392° and with five in 1945 was 7.372°, the difference being only 0.020°. This small difference is not statistically significant and is probably a chance fluctuation of the adjustment of the instrument.

(4) In order to derive probability limits beyond which it may be said that the stress in the disks definitely has not changed, the actual stress change was assumed to be zero and the standard deviation of 13 instrument adjustments over a seven-year period was determined and found to be 0.0333° and the 3-sigma probability limits, ±0.0999°. If the accidental error of observation is combined with this instrument error, the 3-sigma probability limits are ±0.1014°. The change over seven years' time therefore was not greater than approximately ±0.10° or ±1.3%.

(5) The possible change of ±0.10°, equivalent to ±0.30 millimicron, and within the experimental error of measurement, is too small to have the slightest effect on the utility of the standards in use by the glass manufacturers. This experimental error and possible change in stress is too great, however, for drawing broad conclusions concerning the stability of glass at room temperatures with respect to annealing stress.

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HARTFORD-EMPIRE COMPANY
HARTFORD 1, CONNECTICUT

* This possibility is indeed remote because a 2.6% change in seven years could occur only by a linear change starting from an actual value 1.3% higher than the central line in 1938 and passing through a value 1.3% below the central line in 1945. The chance of +1.3% in 1938 is only about one and one half in a thousand and that for a −1.3% in 1945 is the same. But the chance of both events occurring is only about two in a million. Perhaps this is pushing statistics a bit too far, but in any case the rate of decrease, if any, is probably not constant but rather decreasing, so that any decrease in the past seven-year period is greater than that for any future seven-year period.

PERMANENCE OF DISANNEALING STRESSES IN GLASS*

BY F. W. PRESTON

ABSTRACT

Nonmathematical considerations indicate that residual annealing stresses in glass are permanent although Ghering and Green do not draw this positive conclusion from their analysis of measurements over a seven-year period.

The rather loose talk about commercial silicate glasses at room temperature being "undercooled liquids" has led from time to time to bizarre speculations on their rate of flow or change of shape with the passing years. I am assured that the term "undercooled liquid" really means something to physical chemists and that it is unobjectionable if understood as they understand it; but the term is merely a "tale told by an idiot, full of sound and fury, signifying nothing" to the rest of the human race.

In their article, Green and Ghering[1] report the results of a project suggested to them some seven or eight years ago by the Standard Testing Procedure Committee of the Glass Container Association, namely, to make sure that the standard disks for estimating annealing in bottles did not change significantly with the years and were unlikely to change much with the centuries.

These disks are "disannealed" to the extent of about 23 mμ relative retardation near the edge of the disks in a light path of 0.092 in. This is about 0.43 wave length of green light in an inch of light path, and corresponds to a permanent stress of about 510 lb. per sq. in. It corresponds also to a "frozen constitution" distinctly less dense than that of optically annealed glass. Assuming a density of "fully" annealed glass to be 2.5 gm. per cc., the disks probably fall short of this by 0.005 gm. per cc.

On the grounds that the stress is substantial, those who take the undercooled liquid theory too seriously might fear some change as the generations pass away.

* Received June 21, 1945.

[1] L. G. Ghering and T. D. Green, "Annealing Strains in Glass and Their Degree of Permanence," this issue, pp. 288–95.

And in a more modern guise, as discussed by Winter[2] and others, this fear might take the form of a dread of "instability" of constitution, for the disks are very far indeed from that "stability" postulated as essential in some recent writings.

We have then, in these disks, samples of glass under substantial stress and in a low-density form postulated as unstable and therefore more likely to change with the years than "well-annealed" glass subjected to a corresponding load from external sources, such as rods or laths subjected to bending. It would seem that the disks should form good test objects for determining the permanence, or lack of permanence, of the shape of glass objects at room temperature.

For this reason, the Standard Testing Procedure Committee of the G.C.A. decided to go beyond the immediate needs of the glass manufacturers and recommended that a polarimeter of the Adams and Goranson type be constructed and used for checking the disks. The Hartford-Empire Company bore the expense both of the instrument and of much of the work with it as well as the whole cost of making the disks. The instrument was (as far as known) the second of its kind. It is believed that there are now one or two others in existence.

As we now see it, it might have been better to have gone to even greater expense and made an even more ambitious instrument in an attempt to wring the last possible iota of precision and accuracy out of the method. Perhaps that may be done after the war. Ghering and Green have reported faithfully what they found and have analyzed their findings by the best mathematical (statistical) methods known to them. They are clearly remorseful that that last iota of precision is lacking; they are commendably cautious in not drawing any conclusions that are mathematically unwarranted; in particular they refuse to say that there has been *no* change in the disks, though it is abundantly clear that they *found* none.

The present writer is inclined to go at least one step further than Green and Ghering. Their studious analysis shows that the disks have not changed by more than $\pm 1.3\%$ in seven years. This is the gist of the *mathematics*. But as a matter of *physics*, nobody expects an increase anyway. Unless the basic laws of thermodynamics have been suspended and the universe is winding up instead of running down, it is inconceivable that the stresses should *increase* with time. Nor does it seem at all likely that stresses might decrease, or remain unchanged, while *birefringence* (which is what Ghering and Green really measured) increased.

Therefore, if as a matter of pure mathematics the change in stress is certainly limited to $\pm 1.3\%$, as a matter of physics it is very much more restricted. And having this in mind, I feel that common sense must take precedence of both physics and mathematics and confirm us in a belief that, as far as the mere passage of time is concerned, there will be no change in the disks in thousands, or perhaps millions, of years.

The *experiments* do not absolutely prove this, but the conclusion is hard to avoid as a working hypothesis.

In this belief, I am fortified by a private communication from Littleton.* Suspecting that he might have made some experiments on the subject, probably by an entirely different route, I asked his results. He assures me he also has tried and utterly failed to detect any viscous flow at room temperature or for some little distance above that temperature.

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[2] Aniuta Winter, "Time Problem in Annealing," *Jour. Amer. Ceram. Soc.*, **27** [9] 266–74 (1944); "Structural Homogeneity in Glass," *ibid.*, **26** [8] 277–84 (1943).

* J. T. Littleton, Corning Glass Works, Corning, N. Y.

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